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THE SOCIETY OF DYERS AND COLOURISTS
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Holland

Dr. H. J. Selling— *Die Theorie der Farbmessung und die Voraussage von Farbrezepten.*

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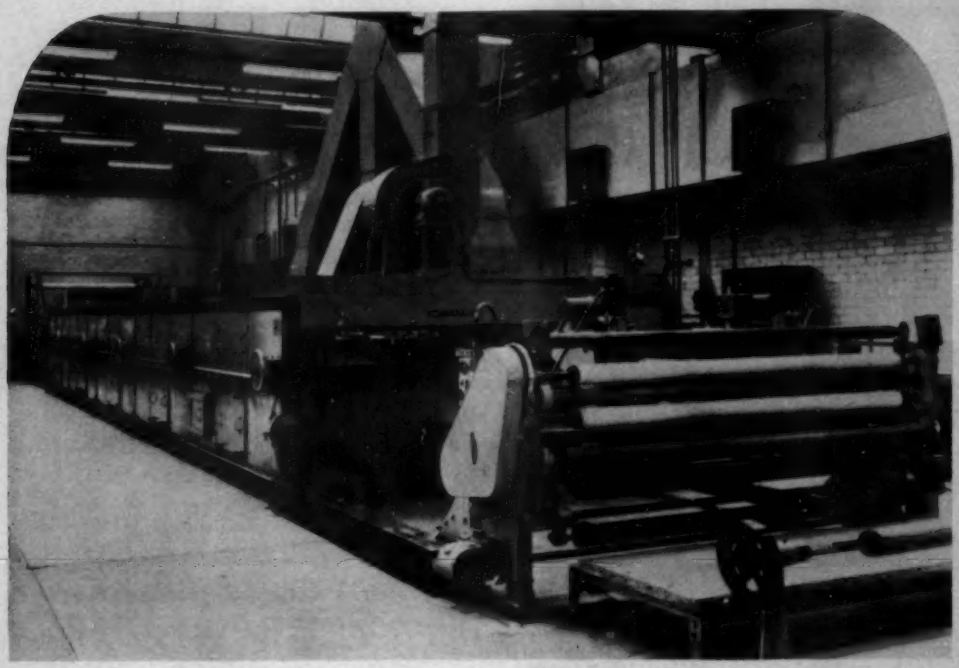

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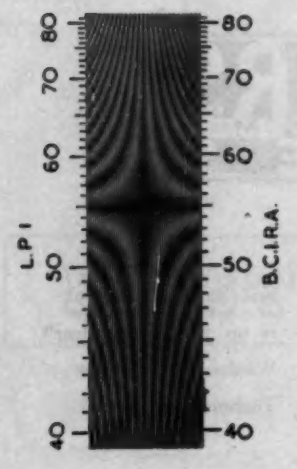

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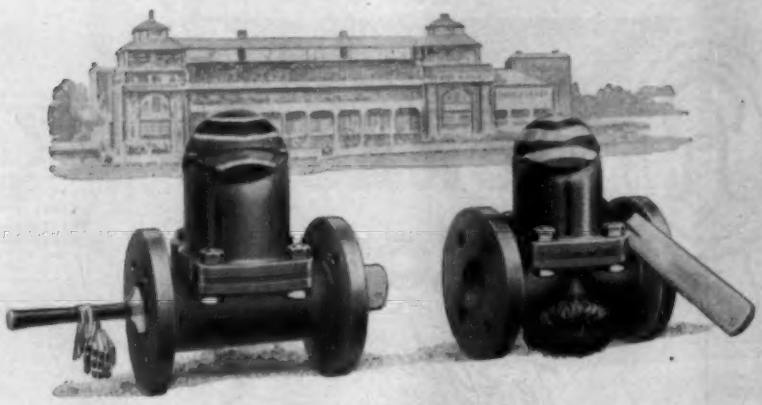

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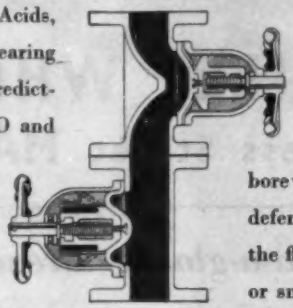

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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

F.T.C.C. PUBLICATION

Influence of Temperature and Humidity on Fading — *P. W. Cunliffe*

COMMUNICATIONS

Spectrophotometric Studies on Metal–Dye Complexes—I — *R. B. Bentley and J. P. Elder*

Photochemistry of Dyes on Proteins and other Substrates — *J. W. Cumming, C. H. Giles, and Miss A. E. McEachran*

Influence of Histological Factors on the Attack of the Wool Fibre by Various Reagents — *R. L. Elliott and J. B. Roberts*

Photochemical Oxidation of Wool in the Presence of Fluorescent Compounds — *D. R. Graham and K. W. Statham*

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Disperse Dyes on Hydrophobic Fibres — *C. L. Bird*

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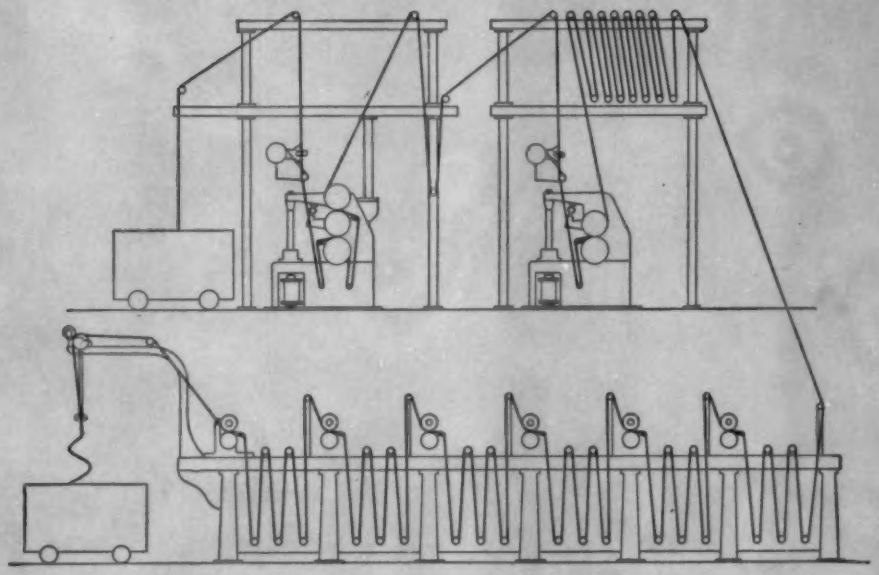

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THE JOURNAL

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Volume 72 Number 6 JUNE 1956 *Issued Monthly*

Proceedings of the Society

The Dyeing of Orlon and Orlon Mixtures

I. M. S. WALLS

Meeting of the Midlands Section held at the King's Head Hotel, Loughborough, on 5th October 1955, Mr. C. A. Mills in the chair

The physical characteristics of the acrylic fibre Orlon are described briefly. Disperse dyes, basic dyes, acid dyes applied by the cuprous ion technique, and indigoid and thioindigoid vat dyes can be used to dye this fibre, and the dyeing and fastness properties of each class of dye are described fully. The selection of dyes for specific purposes is discussed, and some advice on practical dyeing and finishing procedure included. The dyeing of mixtures of Orlon with wool, nylon, and cellulosic fibres is described, and the paper concludes with some comments on the dyeing properties of analogous European acrylic fibres.

During the past year dyers in many parts of the country have been requested for the first time to dye Orlon. Generally only sample lots were processed twelve months ago, but many dyers now regard Orlon dyeing as regular business, and the fibre is being dyed in considerable quantities in forms ranging from loose stock to garments. An accurate estimate of the quantity of this fibre likely to be dyed in the United Kingdom in 1956 cannot be made, but it is probable that it will reach several million pounds weight, and consequently it is appropriate that at this time the dyeing properties of the fibre should be considered in some detail.

Orlon is one of the original members of the acrylic group of fibres and consists essentially of a polymer of acrylonitrile, although a small proportion of a vinyl ester is included in the polymer mix. It is manufactured by E. I. du Pont de Nemours & Co. Inc. in the U.S.A., and is the acrylic fibre best known in the United Kingdom. Similar fibres are manufactured in Europe by, among others, Farbenfabriken Bayer (Dralon), Phrix A.G. (Redon), Rhodiacéta S.A. (Crylor), and N.V. Kunstzijde-Spinneri Nyma, Holland (N 53), but the production of these fibres does not yet approach the figure for Orlon. Orlon is available both as continuous-filament yarn (Type 81) and as a staple fibre (Type 42), but only the latter is particularly important as a textile fibre. A small section of this paper refers to Orlon Type 81, the Type 42 being under discussion in the remainder.

Orlon staple has exceptional bulking power and a warm, soft, lofty handle. It is these properties which are responsible for its tremendous popularity for the outerwear hosiery trade, where it is gaining renown in sweaters or twin sets with a cashmere-like handle. To accentuate this use du Pont have recently introduced "High-bulk Orlon", which contains a proportion of hot-stretched fibre blended intimately with the normal fibre: in boiling water the stretched fibre shrinks, causing the normal fibre to buckle and give a very voluminous, lofty yarn. The abrasion resistance of Orlon is only slightly better than that of the natural fibres, and it is much inferior to nylon and Terylene in this respect. It is not responsive to heat-setting treatments, but pleats which are relatively durable can be introduced into Orlon and Orlon mixture fabrics, while, since the fibre is weaker than Terylene, these materials do not "pill" to the same extent. The shrinkage of Orlon materials in boiling water is very slight (3–5%). As with other synthetic fibres, the moisture regain is low (ca. 1·5% at 70°F. and 65% R.H.), and consequently fabrics are quick drying. Apart from the use already mentioned, blends of Orlon with wool or cellulosic fibres are being increasingly used in both knitted and woven goods, and many developments in these fields may be expected.

DYEING PROPERTIES OF ORLON

Orlon should be dyed either at 95–100°C. or in high-temperature equipment at ca.110°C. When it is important to preserve the lofty handle of the material, e.g. when dyeing knitting yarns, high-temperature dyeing should not be employed. Orlon staple should never be dyed at temperatures much

above 110°C., since excessive shrinkage then takes place. Four classes of dyes may be applied to the fibre, viz. disperse dyes, basic dyes, acid dyes, and indigoid and thioindigoid vat dyes, and these are discussed individually below.

Disperse Dyes

Disperse dyes are readily applied to Orlon by dyeing at 95–100°C. in the presence of a dispersing agent such as Lissapol C, and since they dye the fibre slowly and migrate reasonably well, level dyeings are readily produced. Unfortunately, their exhaustion on to Orlon is rather low, and they do not build up well on this fibre. This deficiency will be readily understood when it is appreciated that the saturation uptake of disperse dyes by Orlon (a figure which may be obtained by dyeing for, say, 48 hr. with 100% dye on the weight of material) is very low. Figures for a few dyes are given in Table I, from which it can be seen that Orlon has a much lower capacity for disperse dyes than has acetate rayon or nylon.

TABLE I

Saturation Uptake (%) of Disperse Dyes at 95°C.

Dye	Orlon	Nylon[1]	Acetate Rayon[1]
Dispersol Fast Yellow G 300	1·4	4·8	7·4
Dispersol Fast Yellow A 300	3·0	5·0	16
Dispersol Fast Orange G 300	1·1	1·8	7·3
Duranol Red 2B 300	1·8	4·5	11·0
Duranol Brilliant Blue CB 300	3·5	8·0	10·5
Duranol Blue Green B 300	1·0	9·5	10·8

These low saturation values indicate that individual disperse dyes cannot be built up to heavy dyeings even by dyeing at a high temperature, although at 110°C. such depths can be produced by using mixtures of selected dyes, since the components, to some extent, build up independently. Medium depths can be produced at the boil by using selected dyes which combine a comparatively rapid rate of dyeing with high tinctorial power and/or a reasonable saturation value. Dyes which may be recommended for these depths are—

Dispersol Fast Yellow A	Duranol Red 2B
Dispersol Fast Yellow G	Duranol Violet 2R
Dispersol Fast Orange G	Duranol Brilliant Blue CB

The low exhaustion of disperse dyes on to Orlon is apparent even with the paler colours, but here it is sometimes an advantage, since it assists the production of level results. The easy production of level dyeings in pale colours with disperse dyes is very important, since basic dyes (the most commonly used alternative class) are very difficult to apply uniformly at such depths.

Disperse dyes, in general, have good fastness properties on Orlon, being more than adequate in this respect for most end-uses of the fibre. Repeated washing at 60°C. has little effect on them, they are fast to burnt gas fumes, while their light fastness even in the pale colours for which they are used is comparatively high. No difficulty is experienced in producing a range of such depths of light fastness 5 to 6, examples being given in Table II. A more variable property is the fastness to pleating treatments, in particular steam pleating. It is important that the dyed Orlon in blends should not mark off on to the other fibres present during pleating, and disperse dyes must be selected with some care if this is to be avoided. Table II illustrates the properties of four dyes which are poor in this respect and six which are satisfactory.

TABLE II

Daylight Fastness and Fastness to Steam Pleating of Disperse Dyes (Pale–Medium Depths)

Dye	Daylight Fastness (*B.S.* 1006)	Fastness to Steam Pleating (10 lb./sq. in. for 15 min.)	
		Effect on Pattern	Staining of Adjacent Material
Dispersol Fast Yellow G ...	5–6	4–5	3
Dispersol Fast Orange G ...	6	3	2
Duranol Red 2B	6	3	2
Duranol Violet 2R ...	6	3	2
Dispersol Fast Yellow A ...	5	5	5
Dispersol Fast Orange B ...	6–7	5	4–5
Duranol Red X3B ...	5–6	4–5	4–5
Duranol Brilliant Violet BR	6	4–5	5
Duranol Brilliant Blue CB	4–5	4–5	5
Duranol Blue Green B ...	5–6	5	5

Pleating fastness is, of course, a less important consideration when selecting dyes for Orlon than for Terylene, since at present most of this acrylic fibre is used for fabrics which will receive only a mild heat treatment during the finishing processes. Good light fastness and comparatively good building-up properties, therefore, will be the criteria by which disperse dyes are generally judged.

Basic Dyes

In the dyeing of Orlon basic dyes are complementary to disperse dyes in many respects, since they are mainly used for the heavier dyeings and possess less satisfactory properties in pale colours. They do not dye Orlon appreciably below 85°C., but as the temperature is raised to 100°C. rapid absorption on to the fibre surface takes place, although diffusion into the fibre is slow and can be significantly accelerated only by raising the temperature to 110°C. The rate of diffusion at 100°C. is not great enough to enable complete penetration to be achieved in a 1–2 hr. dyeing time, and cross-sections of the dyed fibres clearly show ring dyeing. This does not affect the wet fastness properties, which are very good, but it limits the depth of colour obtainable at 100°C., since the quantity of dye which can be absorbed by the fibre is limited to the amount required to saturate the outer ring. If larger quantities of dye are added to the bath the depth achieved is not significantly increased, and, of course, the dyebath exhaustion is progressively reduced. The tinctorial power of

these dyes is so high, however, that only very deep colours such as full blacks and heavy navy blues are unobtainable at 100°C. If dyeing is carried out at 110°C. the rate of diffusion of the dyes is so increased that complete penetration of the fibre is achieved within 30 min., and consequently even the heaviest dyeings are obtainable.

The high initial rate of dyeing on the fibre surface frequently leads to unlevel results with pale colours, and the dyeing should be retarded by adding a cationic agent such as Lissolamine A to the dyebath. Since, in addition, the light fastness of these dyes in pale colours is generally inferior to that of disperse dyes, basic dyes are normally used for such dyeings only when the colours are too bright to be produced with disperse dyes.

It has already been stated that the wet fastness properties of basic dyes on Orlon are very good, and severe washing treatments have very little effect on them. Most of them have excellent fastness to pleating treatments, and their light fastness is surprisingly good on this fibre. A range of dyes of light fastness 4 or better can be selected from the older type of basic dyes (Table III), and new basic dyes are now available with a light fastness of 6 or better[2].

TABLE III

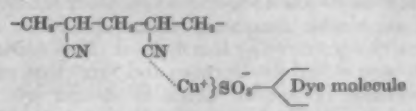
Light Fastness of some Basic Dyes of the Older Type

Dye	Daylight Fastness at Medium Depth (*B.S.* 1006)
Magenta PN	4
Acronol Sky Blue 3G ...	4–5
Indine Blue 2RN ...	4–5
Brilliant Green YN ...	4–5
Malachite Green AN ...	4

Basic dyes should be applied from baths containing 1% of acetic acid (30%), with a cationic retardant if necessary, and medium and heavy dyeings should always be "soaped" after dyeing with a solution of an agent such as Lissapol C at 60–70°C., to remove loose surface dye and improve the rubbing fastness.

Acid Dyes

Acid dyes may be applied to Orlon by the "cuprous ion method". This ingenious technique was originally devised[3] for dyeing dynel, a copolymer of acrylonitrile and vinyl chloride manufactured by the Union Carbide & Carbon Corpn. (U.S.A.), but it may be used to apply acid dyes to any fibre which contains nitrile or cyano side-groups attached to the fundamental carbon chain of the fibre molecule. It depends upon the capacity of cuprous ions for forming co-ordinate valency bonds with nitrile groups as in the inorganic cuprocyanides. Cuprous ions are formed in the dyebath by reduction of a cupric salt, usually copper sulphate, and become attached to the fibre by these co-ordinate bonds. There they form positively charged sites, to which dyes can become attached through their negatively charged sulphonic acid groups. The exact nature of the chemical link between the cuprous ion and the fibre is not known, but a gross oversimplification of the final picture is as follows—



Various reducing agents have been employed in this technique, but probably the most satisfactory are hydroxylamine sulphate[4] and, more recently, metallic copper. Sodium bisulphite can be used when dyeing is carried out in high-temperature equipment. The use of metallic copper has been patented by Sandoz Ltd. and forms the basis of their Sandocryl process[5]. It is a milder reducing agent than hydroxylamine sulphate and consequently generates cuprous ions more slowly, thus making it easier to produce level dyeings, always a difficult task when hydroxylamine sulphate is used. As a milder reducing agent it also shows little tendency to reduce azo dyes, whereas hydroxylamine sulphate sometimes leads to rather dull colours owing to its more vigorous action. To avoid reduction of dye by hydroxylamine sulphate the fibre should be precoppered at 95–100°C. for 15 min. and the temperature reduced to 75–80°C. before the dyes are added. The high initial reduction potential of the dyebath will have fallen by then, and little reduction of dye takes place. Metallic copper, which is used either as strips or as a mesh, is not suitable for all types of dyeing equipment, however, and the full potential value of the Sandocryl process cannot yet be assessed. Whatever reducing agent is employed, the dyebath pH should be between 2 and 3 to ensure formation of the requisite number of cuprous ions.

Not all acid dyes are suitable for application by the cuprous ion method, and only a comparatively small number have good dyeing properties. Many of these are simple monosulphonated acid levelling dyes, the more highly sulphonated dyes of this class generally being much poorer in this respect. Selected acid milling dyes also are suitable, particularly when dyeing is carried out at a high temperature. The colours produced are similar to those given on wool by a normal dyeing method, indicating that no copper complex is formed between the dye and the cuprous ions. When chrome dyes are applied by this method they yield the colour of the unchromed dye on wool. The light fastness of acid dyes on Orlon is similar to that on wool, but the wet fastness on Orlon is much superior, the dyes being unaffected by repeated washing at 60°C. In this respect acid levelling dyes are just as fast as the milling dyes.

Since disperse and basic dyes can be applied to Orlon so easily, and since their fastness properties on this fibre are so good, it would seem unlikely that the cuprous ion method will ever be very extensively used. Even when copper is used as the reducing agent, the dyeing process is considerably more complicated than is the application of disperse or basic dyes, and, since no levelling takes place, level results, particularly in the lighter depths, are

not readily obtained. The process has advantages for the production of very deep colours, which are not readily produced with basic dyes, and can be suitably used when dyeing loose stock. Colours such as blacks and navy blues, however, are expensive when dyed by this method. Furthermore, it has been stated by Dorman and Fox[6] that some degradation of acrylic fibres is caused by the cuprous ion method if it is followed by certain steaming operations during finishing. Turnbull[7] states more specifically that copper accelerates the browning of Orlon fibre that normally results from excessive drying times or temperatures, and indeed it has been found that undyed, coppered Orlon can turn brown during a comparatively mild steaming treatment such as the postboarding of half-hose.

Vat Dyes

Selected indigoid and thioindigoid vat dyes can be applied to Orlon by dyeing as the alkaline leuco form at ca. 95°C. in the presence of Formosul at a pH of about 10. The dyes are vatted in the usual manner, and the pH of the dyebath is reduced to the required level with sodium bicarbonate. The dyes are developed by a chemical oxidation with e.g. sodium perborate or percarbonate at 95°C. Some of the dyes which can be applied in this way and built up to heavy dyeings are listed in Table IV, from which it can be seen that their light fastness is very high.

TABLE IV

Light Fastness of some Durindone Dyes

Durindone Dye	Daylight Fastness at Medium Depth (*B.S.* 1006)
Scarlet 2B ...	6–7
Pink FF ...	6–7
Red 3B ...	7
Brown G ...	7
Blue 4BC ...	6–7

The wet fastness of these dyes, as would be expected, is excellent, and they have good fastness to any heat treatments which may be given to the fibre. Consequently they provide a narrow range of dyes capable of giving dyeings of excellent all-round fastness, and as such will have definite though limited uses for this fibre. It is unlikely that they will be widely used, since they are expensive and their excellent fastness properties will rarely be required on a fibre which will be used principally for materials where a rather lower level of wet fastness is acceptable.

Indigo can be applied to Orlon as either the acid or the alkaline leuco derivative, while the Alcian dyes can be used to produce pale colours of excellent fastness properties. Most Soledon dyes can be applied in pastel colours by a pad–development process.

BULK DYEING

One of the most valuable properties of Orlon staple fibre is a very soft, lofty handle, and for most end-uses of the fibre it is particularly important that this handle should not deteriorate as a result of the dyeing process, and that it should be accentuated by subsequent finishing treatments. Generally, therefore, Orlon yarns and knitted fabrics should be dyed in a completely relaxed condition, all tension in a hot, wet state being avoided.

Yarn Dyeing

The material is preferably dyed in hank form, the hanks being completely submerged in the dye liquor. They may be looped or chained loosely together and dyed in hosiery bags in paddle machines, or dyed in cans which are packed as loosely as possible commensurate with level dyeing. More conveniently, yarn may be dyed in machines such as Hussongs if certain precautions are taken. Sampling should be carried out on an isolated hank, which should be withdrawn from the machine without raising the rest of the material, since frequent lifting of the hot yarn will spoil its handle owing to extension by the weight of liquor absorbed. For the same reason the yarn should be cooled slowly to 70–75°C. after dyeing before it is lifted from the dyebath. Hank dyeing in a cascade machine is unsatisfactory, and package dyeing is not generally recommended, as the handle of the material produced compares unfavourably with the handle of yarn dyed in a fully relaxed state. If some deterioration of the handle can be tolerated, regular Orlon can be dyed in packages provided that they are wound under low tension.

High-bulk Orlon must be treated with even greater respect, and it should not be dyed in packages. This material should be treated in boiling water without any liquor circulation to induce shrinkage in the stretched portion of the fibres at this stage, cooled slowly, and then scoured and dyed.

Garment and Piece Dyeing

Garment dyeing presents few problems, and is probably the most suitable way of dyeing Orlon when it is to be used in the outerwear hosiery trade. As with yarn, the garments should be cooled slowly after dyeing. A softening agent such as Velan PF may then be applied, and the garments tumbler-dried with live steam blown in during the latter stages of this process. This procedure will lead to material of a most satisfactory loft and soft handle. The garments should not be pressed or boarded.

If knitted pieces are dyed on the winch, some deformation of the fabric and creasing will take place owing to the extension of the material in a hot wet state. A more suitable, although more tedious, method involves cutting the fabric into comparatively short lengths, folding it loosely into cubes, and dyeing in hosiery bags. Some woven fabrics may be dyed on winches without deterioration of the handle, although they also are very prone to creasing and rope-marking.

ORLON MIXTURES

It is likely that Orlon staple fibre will be extensively used in blends with other fibres, in particular with wool and viscose rayon staple, and

a brief description of the dyes and dyeing methods used for these blends is therefore included in this paper.

Orlon–Wool Blends

These blends are becoming increasingly widely used, since they give soft, bulky yarns very suitable for knitted garments, and, provided that a reasonable proportion of Orlon is used, say 40–50%, they have an advantage over all-wool in that they shrink very little on washing. The special advantages of Orlon–wool in woven fabrics are less obvious, but the properties of such materials are being thoroughly investigated, and it is not unlikely that they, too, will be found to have a special combination of virtues which will render them desirable for some end-uses.

There is little variety in the dyeing methods available for Orlon–wool blends. Basic dyes should be used to dye the Orlon component, since acid dyes, applied by the cuprous ion technique, dye the wool preferentially, and disperse dyes stain the wool at least as heavily as they dye the Orlon. Basic dyes are absorbed by wool, but if dyeing is carried out at 95–100°C. in the presence of 2–3% glacial acetic acid (calculated on the total weight of the goods) and Lissapol N (2 g./litre), the basic dyes are gradually transferred to the Orlon until the wool is only lightly stained. The depth of this stain and the time required to promote the necessary transfer of the dye to the Orlon vary with the depth dyed, the dyes used, and the dyeing temperature, but with selected dyes at 98°C. a dyeing time of 60–90 min. is sufficient to reduce the staining to a minimum. If the quantity of acetic acid is reduced or the Lissapol N omitted, the transfer of the dyes from the wool is slower, and heavier staining will be observed. It must also be remembered that Orlon can absorb only limited amounts of basic dyes in 90 min. at the boil, and that if excessive quantities are used when trying to produce heavy dyeings the excess dye will be absorbed by the wool and cause heavy staining. Very heavy dyeings, therefore, should be avoided when possible.

The wool may be dyed in several ways, but acid levelling or acid milling dyes will be most commonly used. The wet fastness properties of acid milling dyes are more appropriate here, since basic dyes have such good fastness on Orlon. In the simplest dyeing method the Orlon is dyed first and the wool cross-dyed in the same bath with acid levelling dyes. Alternatively, the Orlon is dyed first, the material is rinsed and neutralised with ammonia, and acid milling dyes are applied to the wool. These methods are convenient, since in, for example, an intimate 50 : 50 blend the colour produced on the Orlon component need only approximate to the desired colour, accurate shading being carried out on the wool, which is much easier than shading the Orlon. The third method is to dye the wool with milling dyes, completing their exhaustion by adding the full quantities of acetic acid and Lissapol N required for dyeing the Orlon, adding the basic dyes to the same bath at about 80°C., and boiling for a further 60–90 min. This is a rather shorter process, and it avoids the neutralisation stage required when the Orlon is dyed first and milling dyes are to be used subsequently on the wool, but the transfer of the basic dye to the Orlon is not so readily followed and shade matching is rather more difficult.

Blends of Orlon with Cotton or Viscose Rayon

The Orlon component of such blends may be dyed with disperse dyes for the lighter colours and basic dyes for the deeper colours. In pale colours there is little staining of the cellulosic component by the disperse dyes, and direct cotton dyes can be applied in the same bath. With medium depths the stain on the cellulose may be removed by a scour at 60°C. or a "permanganate clearing treatment" prior to cross-dyeing with direct dyes, vat dyes, or Soledons as required. When basic dyes are used, they should be applied to the blend by the method used for dyeing 100% Orlon. If excessive quantities of dye are avoided, the staining of the cellulosic portion is not heavy and may be further reduced by a scour at 60°C. The cellulosic portion may then be filled in as desired.

Orlon–Nylon Blends

Most of the statements made on the dyeing of Orlon–wool blends are applicable to Orlon–nylon blends, but it is not necessary to use Lissapol N, nor to employ more than 0·5–1·0% glacial acetic acid with the basic dyes in order to promote transfer of the dye to the Orlon.

CONTINUOUS-FILAMENT ORLON (TYPE 81)

The fibre molecules in continuous-filament Orlon are much more highly oriented and tightly packed than those in the staple fibre, and consequently dye molecules will penetrate the filament yarn very much more slowly. Since the staple fibre cannot be satisfactorily dyed below 95–100°C., it will be obvious that the filament variety will be extremely difficult to dye, and in fact only pale colours can be produced at temperatures up to 100°C. The diffusion of dyes into the filament can, of course, be accelerated by dyeing at higher temperatures, and medium depths can be produced with disperse dyes and dark colours with basic dyes by dyeing Orlon 81 at 120–125°C. Since it is virtually impossible to dye Orlon 81 in normal equipment, this fibre will have very limited textile uses.

EUROPEAN ACRYLIC FIBRES

Several European fibre producers have developed acrylic fibres which are very similar in both fibre properties and dyeing properties to Orlon staple, e.g. Pan (CFM) and those mentioned on p. 261. Chemically these fibres are closely related to Orlon, and differences between them in respect of dyeing and fastness properties may be resolved into—

(*a*) Differences in rates of dyeing
(*b*) Differences in the maximum depths that can be produced
(*c*) Differences in light fastness.

The differences between the rates of dyeing of the various staple fibres are not large enough to be

important, although some of the fibres dye rather more slowly than Orlon 42. However, there are very significant variations in the maximum depths that can be produced with some dyes when dyeing at 100°C. for 1–2 hr., these variations being particularly important with basic dyes, which are used to produce deep colours. Generally speaking, these dyes can be built up to heavier depths on Orlon than on the other fibres, while smaller variations are apparent among them. It is particularly difficult to produce heavy dyeings on Redon with basic dyes. Variations in the light fastness of dyes on the different fibres also are important. Generally, dyes show the highest light fastness on Orlon and Dralon, while their fastness on the other fibres is to varying extents poorer. The light fastness of basic dyes does not vary greatly (up to, perhaps, 1 point on the S.D.C. 1–8 scale), but the fastness of disperse dyes falls off considerably on Redon and N 53 (sometimes 1–2 points) and even more on Pan. Consequently, disperse dyes which are used for pale colours of good light fastness on Orlon are quite inadequate for this purpose on some of the other fibres. Therefore the cuprous ion method, which can be used to produce both heavy dyeings and high light fastness, will probably be more widely used with some of these fibres than with Orlon.

* * *

To conclude this paper I would like to express my thanks to Mr. B. Batterbee, who has carried out the experimental work upon which the paper is based.

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Discussion

Mr. C. A. MILLS: The cuprous ion technique is certainly a novel approach to dyeing. Can the lecturer say whether it was an ingenious invention or an accidental discovery?

Dr. WALLS: While it would be pleasant to think that the method was born entirely as the result of deep and acute thought, it has been rumoured that it was discovered through a dyeing carried out in a dirty dyepot containing copper; but even if the latter is true, the "inventors" deserve credit for the way in which they turned this accident to good effect.

Mr. W. RUSSELL: What is the best way to ensure fastness to rubbing on Orlon?

Dr. WALLS: Medium and heavy dyeings, whatever the dyes used, should always be scoured after dyeing, 15–20 min. at about 60°C. in a Lissapol C solution being recommended.

Mr. J. RAYMENT: Will the lecturer enlarge upon his remarks on the properties of disperse dyes on Orlon, particularly with regard to independent build-up?

Dr. WALLS: It has been known for some time that mixtures of disperse dyes build up better than the individual components. For example, a blue dye which consists of a mixture of two components builds up better on, say, acetate rayon than does either of the components applied separately. Schuler and Remington [8] observed this phenomenon on Terylene (Dacron). With most fibres, however, this property is of importance only with regard to the rapidity with which heavy dyeings can be produced, since the saturation uptakes of most disperse dyes are comparatively high and rarely constitute a practical limit to the depth of colour that can be produced. With Orlon, however, where the low saturation values seriously limit the depths obtainable with single dyes, it is a great advantage to use mixtures of dyes and thus increase the total quantity of dye that can be absorbed. The extent to which dyes build up independently in mixtures depends on their chemical structures: if these are very similar, the improvement effected will not be large; if they are completely different, e.g. an azo and an anthraquinone dye, the total amount of dye which can be absorbed may approach the sum of their individual saturation values [8].

Dr. A. DATYNER: Why is the easily dyeable type of Orlon, i.e. 42, not obtainable in filament form?

Dr. WALLS: Orlon 42 staple fibre is closely related chemically to Orlon 81 filament yarn. The dyeing difficulties associated with the latter are mainly due to the high degree of orientation and the consequent tight packing of the fibre molecules in this yarn, which has presumably been subjected to a much more severe drawing process than the staple fibre. The close packing of the fibre molecules causes dye molecules to find the greatest difficulty in penetrating the yarn.

Mr. H. W. PARTRIDGE: The lecturer has told us how to get dyes on to Orlon. Can he tell us how to get them off?

Dr. WALLS: Some basic dyes and azo disperse dyes can be stripped with acid Formosul; generally, however, acid sodium chlorite is required. If, during a dyeing with disperse dyes, the shade comes up too heavy, sufficient dye can usually be removed by treating at the boil in a fresh bath containing Lissapol C. Unfortunately, if basic dyes or acid dyes are being applied by the cuprous ion method and the shade is overshot, chemical stripping is required.

Mr. A. W. CARPENTER: Has the lecturer anything to add to published information on the bleaching of Orlon?

Dr. WALLS: No. Orlon staple as manufactured is quite a good white, which can be improved by bleaching with oxalic acid; the best bleach, however, is obtained by using acid sodium chlorite. Other bleaching agents have little effect.

Mr. T. H. HEYES: Does Orlon suffer from light degradation in a similar way to nylon?

Dr. WALLS: No. I would add that it is renowned for its weathering properties.

Mr. S. HEGGS: It has been stated elsewhere that some basic dyes on Orlon change their tone in finishing.

Dr. WALLS: Some basic dyes change colour during dry or steam heat treatments during finishing but recover more or less quickly. Perhaps Mr. Heggs is thinking of published information in American journals, where it is recommended that sodium acetate should be used together with acetic acid in the dyebath to exert a buffering action and prevent subsequent colour changes during finishing. I can only say that we have never heard at first hand of any permanent changes which have resulted even when sodium acetate is omitted; and it is difficult to see what effect the addition of sodium acetate can have when the dyeing is in any event being carried out at a pH of round about 5.

A MEMBER: Is it necessary to set Type 81 fabric to ensure stability and guard against creases during dyeing? What is the best way of removing creases once formed?

Dr. WALLS: Since I have very little experience of dyeing Orlon filament yarn in bulk, and none of winch dyeing fabrics of this material, I cannot answer these questions satisfactorily. Only pale colours could be produced on such machines in any event. Some prestabilisation of the pieces by either dry or steam heat will almost certainly be required to minimise creasing, but since the manufacturers state that Orlon is not receptive to heat setting treatments in the same way as is Terylene or nylon, it is doubtful how effective such a treatment would be.

Mr. W. RUSSELL: From examination of the lists of disperse dyes recommended by the lecturer, I get the impression that those dyes which are fastest to sublimation also have the slowest dyeing rate. Is this so?

Dr. WALLS: In general, yes; although there are important exceptions on either side. For example, Dispersol Fast Yellow A is one of the more rapidly dyeing disperse dyes, yet it has good sublimation fastness.

Mr. T. ALLEN: I note that light fastness figures are given as daylight exposures. Is there any anomaly in fading lamp exposures equivalent, say, in the case of basic dyes, to that of the same dyes on cellulose?

Dr. WALLS: No significant anomaly has been observed with basic dyes, but appreciable differences have sometimes been observed with some disperse dyes, fading lamps tending to reduce their apparent light fastness. The magnitude of the effect depends upon such factors as the temperature and the humidity in the lamp, and the distance between the pattern and the lamp; the hotter lamps, for example, have a more severe effect.

Mr. A. G. TYLER: Can the wool in Orlon blends be dyed with chrome or metal-complex dyes? If so, which fibre is dyed first?

Dr. WALLS: Yes. Metal-complex dyes can be applied in the same way as acid milling dyes, and the Orlon can be dyed first or second as described in the text, preferably first. Chrome dyes are rarely used, since their high wet fastness is rarely required, the dyeing process would be lengthened, and the outerwear hosiery trade requires brighter colours. They could be used, however, and the wool should then be dyed first.

Mr. J. SAUNDERS: With reference to shrinkage difficulties encountered in dyeing Orlon yarn in package form, could it not be wetted out, dried, and then rewound for dyeing?

Dr. WALLS: This procedure would enable package dyeing to be carried out without the shrinkage preventing dye circulation. Unfortunately, the shrinkage would still take place during the original boiling off, and since the yarn is not then in a tensionless state it would become stringy, losing some of its softness, loft, and bulk. In particular, the handle of high-bulk Orlon would be ruined.

Mr. T. HEYES: Can uneven yarn stretch or set in preparation cause shade variations?

Dr. WALLS: I have never observed this with Orlon staple yarns. Staple fabrics, of course, are not heat-set before dyeing, so the question does not arise. Filament yarns stretched to different degrees during manufacture will cause shade differences.

Mr. A. P. KERSHAW: I would question the lecturer's statement that he does not envisage any great use of Orlon for hose owing to its abrasion properties. I have seen fine-gauge women's hose which have worn quite well.

Dr. WALLS: I was referring to the use of the staple fibre for half-hose. The filament fibre is stronger, but in view of the difficulty of dyeing it seems unlikely that it will ever find a considerable market in ladies' hosiery.

Mr. KERSHAW: Experimental quantities of fur–Orlon blends have been made in the U.S.A. Has such a blend been tried in Britain?

Dr. WALLS: Not to my knowledge; but I imagine that such blends will be investigated soon, since they are proving popular in America.

Mr. R. GREENLEES: Has any success been achieved with the pad-dyeing of Orlon?

Dr. WALLS: To answer very broadly, laboratory work has shown promise along one or two lines. So far, there has been no demand in this country for such a method.

Mr. C. A. MILLS: Can stripiness occur on Type 81 as on filament nylon?

Dr. WALLS: Most definitely, yes.

Mrs. B. DRAPER: As Orlon is slightly plastic at the boil, does this affect the lustre as with dynel?

Dr. WALLS: No. Orlon does not need any relustring treatment after dyeing corresponding to that given to dynel.

Dr. R. RICHARDSON: Has the lecturer any knowledge of the extent to which dress fabrics are being made from Orlon–wool blends? How are these fabrics best processed?

Dr. WALLS: Knitted fabrics may, of course, be yarn-dyed or winch-dyed. Very little work seems to have been done so far in this country on this blend in woven fabrics. Where it has been used, the fibres seem to have been dyed in loose form, which is certainly preferable from the dyeing aspect. Winch dyeing would be required otherwise, jig dyeing being avoided if possible, since it will tend to give fabrics of an inferior handle.

Mr. A. KEELING: Is there any information on the fastness of disperse and basic dyes on Orlon to dry cleaning?

Dr. WALLS: They are fast to normal dry cleaning treatments, although trichloroethylene at elevated temperatures may remove some disperse dye from a fabric.

COMMUNICATIONS

Interaction between Chlorazol Sky Blue FF and Chrysophenine G in Aqueous Solution

A. N. DERBYSHIRE and R. H. PETERS

The absorption spectra of mixtures of Chlorazol Sky Blue FF and Chrysophenine G in aqueous solution have been measured at room temperature and at 60°c. Spectra of regenerated cellulose sheet dyed with the two dyes from mixture dyebaths have also been determined. Paper and column chromatography on mixtures of the two dyes confirm the formation of complexes, deduced from the spectral measurements. The heat of formation of the 1 : 1 complex of the two dyes, measured by direct calorimetry, is −15 kcal. per mole. Finally, equilibrium adsorption of the two dyes by cotton from mixture dyebaths has been determined.

It is concluded that formation of a 1 : 1 complex occurs in aqueous solution to an appreciable extent even at 90°c. This complex formation explains in a semi-quantitative way the change in dye adsorption compared with that of the same dyes when applied singly. It is very probable that failure of dyeing data on single dyes to predict the behaviour of the same dyes in mixtures is due to interaction of this kind between the dyes in solution.

Introduction

It is well known that the behaviour of direct dyes when applied from a mixture is frequently very different from their behaviour when dyed singly. This effect limits the usefulness of dyeing data in the dyeing of mixtures. Thus, Lemin, Vickers, and Vickerstaff[1] have shown that rates of dyeing and equilibrium exhaustion results cannot be used with certainty to predict compatible combinations. It is not clear from work of this kind whether the effect is primarily due to modification of the rates of adsorption of dyes or of their equilibrium adsorption values. However, Neale and Stringfellow[2] have measured the equilibrium adsorption on cotton of a number of direct dyes in binary mixtures, and their results show that the apparent affinities of the dyes are considerably modified in mixtures. Of the dyes which they examined, the greatest change in adsorption value is found for the pair: Chlorazol Sky Blue FF and Chrysophenine G. In the hope of obtaining more information about the mechanism of the process, this combination has been studied in greater detail.

Neale and Stringfellow showed that the absorption spectrum of the mixture at room temperature did not agree with the sum of the spectra of the individual dyes, and they suggested that the dyes interacted in solution. These measurements have been extended to cover a range of concentrations of each component, and similar measurements have been made at higher temperatures to determine the extent to which interaction is reduced by increasing temperature. The spectra of the two dyes adsorbed together on Cellophane sheet have also been examined. The formation of complexes of the two dyes in aqueous solution, deduced from the spectral measurements, has been confirmed by paper and column chromatography, and calorimetric measurements have been made to give an indication of the magnitude of the heat of complex formation. Finally, the adsorption measurements of Neale and Stringfellow, which were all made in solutions containing an excess of Chrysophenine G, have been extended to include dyeings from mixtures containing excess of Sky Blue FF.

Experimental

1. ABSORPTION SPECTRA

All the absorption spectra of the dyes in aqueous solution were measured by means of the Cary recording spectrophotometer. For work above room temperature the absorption cells were immersed in a cubical water bath of optical glass forming part of the microthermostat unit, which has been fully described before[3]. Formation of air bubbles was eliminated by using freshly boiled-out distilled water in the thermostat, boiling out dye solutions, and making up with boiled-out water

prior to measurement. Solutions of the phototropic Chrysophenine G were subjected to normal room illumination prior to measurement, since the work of Willis *et al.*[4] showed that the change from "illuminated" to "dark" spectrum occurred slowly, while the reverse change was much more rapid.

The absorption spectra of the dyed Cellophane sheets were determined by means of the Hardy recording spectrophotometer. The sheets were soaked in distilled water and pressed between plane glass plates for measurement.

2. ADSORPTION EXPERIMENTS

Equilibrium dye adsorption on cotton was measured at 90°C. Approx. 0·1-g. hanks of cotton yarn were dyed in 75-ml. portions of dye liquor in a thermostat at 90°C. for periods of not less than 24 hr. After being dyed, the yarn sample was removed from the dyebath, washed free from entrained dye liquor, and conditioned at known humidity and temperature. It was then weighed, and the adsorbed dye extracted with pyridine. The dyebaths and stripping liquors were diluted to a pyridine concentration of 25% and the content of dye was determined colorimetrically. Check measurements on solutions of known strength in 25% aqueous pyridine showed that the dyes obeyed Beer's law both singly and in mixture and the spectrum of a mixture was the sum of the spectra of its components. The only difficulty in the method arises from the absorption by Chlorazol Sky Blue FF at the peak wavelength of Chrysophenine G. Correction was made for this from the observed density at the peak wavelength of Chlorazol Sky Blue FF, using the ratio of Chlorazol Sky Blue FF densities at the two wavelengths found by measurements on solutions of this dye alone. However, in those dyeings in which a considerable excess of the blue dye is adsorbed the measurement of the amount of Chrysophenine G adsorbed cannot be made very accurately for this reason.

The dyed Cellophane sheet was obtained by dyeing to equilibrium for 24 hr. in a similar manner to that employed for the measurement of adsorption by cotton. The sheets were removed from the dyebaths and rinsed in cold water to remove dye liquor, and their absorption spectra were measured. They were then conditioned at known humidity and temperature, weighed, and stripped with pyridine for estimation of dye content.

3. CHROMATOGRAPHY

In order to check the deductions made from the absorption spectra, paper chromatography measurements were made on a number of solutions containing varying proportions of the two dyes and also on each dye alone. The dye solutions were made up in aqueous solution without added salt and run out with distilled water. Under these conditions the two dyes singly showed almost equal apparent affinities, each spreading the same distance relative to the spread of the solvent. In each case when a mixture was used a green band was produced at the edge of the chromatogram, showing the presence of a complex of lower affinity than either dye alone. The central region of the chromatogram was generally coloured by the dye in excess in the mixture, except that in solutions of near equality of the two dyes on a molar concentration basis the blue component preponderated in the centre. This effect may be due to the greater colour value of Chlorazol Sky Blue FF.

This work showed clearly that complexes of the two dyes were formed in aqueous solution at room temperature. The production of a green band on the outside edge of the chromatogram, more nearly keeping pace with the solvent than the single dyes, is exactly the behaviour to be expected of a complex of this kind. The high charge of such an anion will considerably reduce its adsorption, particularly under the conditions used, i.e. in absence of added electrolyte.

In the hope of obtaining a sample of the complex and determining its constitution, a column of alumina was set up and mixture solutions similar to those used in the paper chromatography were run through it with distilled water. Separation was not so complete as had been hoped, but analysis of the first sample collected in each case was in agreement with the deductions made from the absorption spectra (see Results section), that a 1 : 1 complex was formed in the presence of excess of Chlorazol Sky Blue FF, while complexes richer in Chrysophenine G were formed when that dye was in excess.

4. CALORIMETRY

The heats of interaction were determined at 25°C. in the calorimeter which has been described elsewhere[5]. The more soluble Chlorazol Sky Blue FF was added in concentrated solution from the mixing pipette to Chrysophenine G solution in the body of the calorimeter, and the heat produced was measured in the usual way. Separate experiments were performed at each concentration used, to determine the heat change due to dilution of the single dyes.

MATERIALS

The dyes used were obtained by purification of commercial dye samples, by the method of Robinson and Mills[6]. The cotton used in the adsorption experiments was a 2/20s bleached yarn, which was scoured in a solution of 2 g. soap plus 1 ml. ammonia (sp.gr. 0·88) per litre. The cellulose sheet, dyed to determine the spectra of the dyes on this substrate, was Cellophane 300. Since only comparative results were required, the Cellophane was given no pretreatment before dyeing.

Results

I— ABSORPTION SPECTRA OF AQUEOUS SOLUTIONS

It is already known[2] that the absorption spectrum of a mixture of these dyes in aqueous solution at room temperature is not the same as the sum of the spectra of its constituents. This non-additivity is illustrated in Fig. 1, in which are plotted the optical densities of solutions of Chlorazol Sky Blue FF and Chrysophenine G, each at 0·02 g./litre, the sum of these two spectra, and

the optical density of a mixture of the two dyes at 0·02 g. of each dye per litre. The change in spectrum is characterised by a shift of both absorption maxima to longer wavelengths, with a decrease in density at the Sky Blue peak and an increase in density at the Chrysophenine G peak.

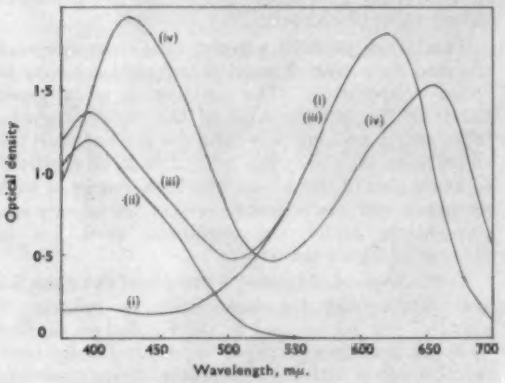

Optical density
Wavelength, mμ.
(i) Chlorazol Sky Blue FF (0·02 g./litre)
(ii) Chrysophenine G (0·02 g./litre)
(iii) Sum of (i) and (ii)
(iv) Mixture of (i) and (ii) (total dye concn. 0·04 g./litre)

FIG. 1— Non-additive Spectra of Chlorazol Sky Blue FF and Chrysophenine G

These spectral changes were then examined more systematically by varying the ratio of the constituent dyes in the mixture. The spectra of the single dyes were also measured over a range of concentrations up to $2{\cdot}5 \times 10^{-5}$ M. for Sky Blue FF and up to $5{\cdot}0 \times 10^{-5}$ M. for Chrysophenine G. Extinction coefficients calculated from these data showed only small random changes from one concentration to the next, and there was no significant departure from Beer's law for either dye. This enabled concentrations in mixture to be chosen fairly freely to give the required ratio of constituents without complicating the interpretation of the results.

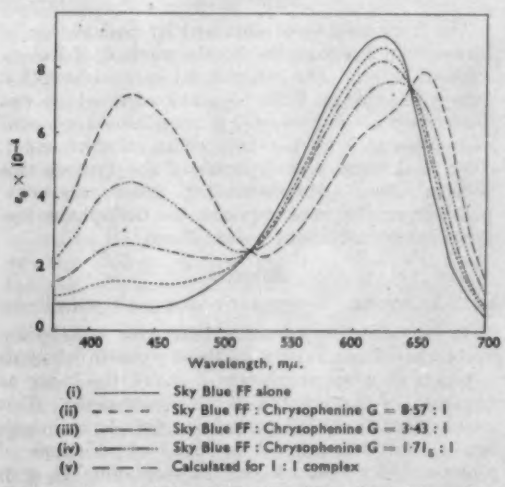

$\epsilon_\phi \times 10^{-4}$
Wavelength, mμ.
(i) Sky Blue FF alone
(ii) Sky Blue FF : Chrysophenine G = 8·57 : 1
(iii) Sky Blue FF : Chrysophenine G = 3·43 : 1
(iv) Sky Blue FF : Chrysophenine G = 1·71$_5$: 1
(v) Calculated for 1 : 1 complex

FIG. 2— Excess of Chlorazol Sky Blue FF at Room Temperature

The spectra of three solutions containing different excess amounts of Chlorazol Sky Blue FF were measured. Extinction coefficients calculated on the concentration of Chlorazol Sky Blue FF from these results are plotted in Fig. 2 (curves (ii)–(iv)), together with the corresponding curve (i) for the blue dye alone. It will be seen that the spectra pass through two well defined isosbestic points, and calculation shows that they are consistent with the hypothesis that under these conditions a 1 : 1 complex is formed, the reaction going virtually to completion, so that all the Chrysophenine G is present in the form of the complex. The calculated spectrum of this complex is also shown in Fig. 2 (curve (v)).

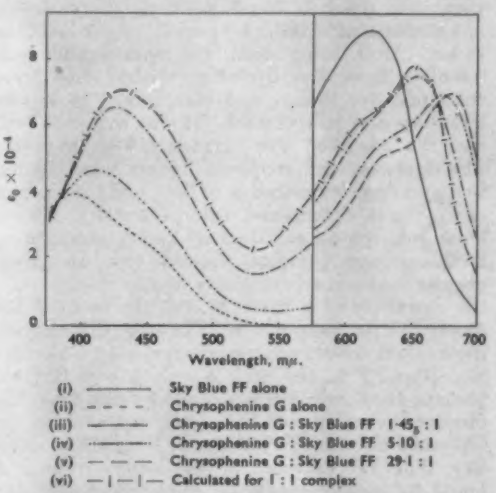

$\epsilon_\phi \times 10^{-4}$
Wavelength, mμ.
(i) Sky Blue FF alone
(ii) Chrysophenine G alone
(iii) Chrysophenine G : Sky Blue FF 1·45$_5$: 1
(iv) Chrysophenine G : Sky Blue FF 5·10 : 1
(v) Chrysophenine G : Sky Blue FF 29·1 : 1
(vi) Calculated for 1 : 1 complex

FIG. 3— Excess of Chrysophenine G at Room Temperature

A corresponding series of measurements was then made with Chrysophenine G in excess. The results are shown in Fig. 3, in which extinction coefficients calculated on the concentration of Chlorazol Sky Blue are plotted from 700 to 575 mμ. and values calculated on the concentration of Chrysophenine G from 575 to 375 mμ. Inspection of the long-wave region of the graph shows that the formation of a 1 : 1 complex does not adequately explain the data under these conditions. The three measured curves all lie on the opposite side of the 1 : 1 complex spectrum to that of the blue dye alone. While this is consistent with the hypothesis that all the Sky Blue is combined with Chrysophenine G, it suggests that complexes richer in the yellow dye are formed in the presence of an excess of this dye. This view is strengthened by the failure of attempts to represent the spectra in the short-wave region as mixtures of those of Chrysophenine G and 1 : 1 complex. It should be noted that in one of the mixtures (curve (v)) accurate measurement at long wavelengths in presence of a very large excess of Chrysophenine G was obtained by use of a mixture containing a high concentration of this dye, taking advantage of its negligible absorption above about 575 mμ. The spectrum of this mixture was consequently

not measurable at shorter wavelengths, and is omitted from this region of Fig. 3.

Thus, the deductions from the spectra at room temperature may be summarised—

(1) A 1:1 complex is formed almost completely

(2) Higher complexes are formed to a certain extent in presence of excess of Chrysophenine G.

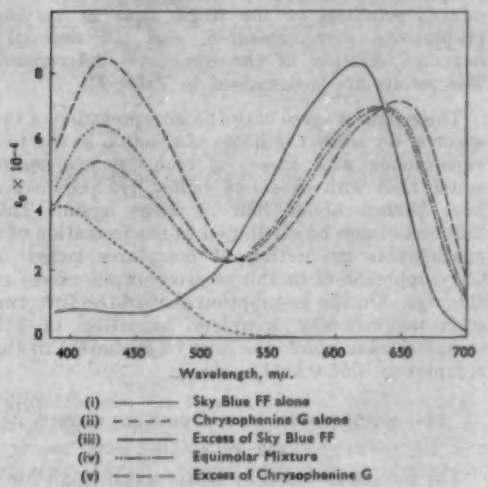


FIG. 4—Absorption Spectra at 60°C.

Spectra measured at 60°C. are shown in Fig. 4. The diagram includes the spectra of the single dyes, together with those of three mixtures, containing excess of Chlorazol Sky Blue FF, equimolar constituents, and excess of Chrysophenine G respectively. These three all pass through a single isosbestic point with the blue dye in the long-wave region, where the effect of uncombined yellow dye is negligible. This indicates that formation of complexes richer in Chrysophenine G does not occur appreciably at this higher temperature. The continuing spectral shift beyond that of the equimolar mixture also shows that formation of the 1:1 complex, while it still persists, is less complete than at room temperature.

It is possible from these spectral changes to calculate a rough order of magnitude for the instability constant K of the complex, according to the mass action equation—

$$K[D_c] = ([D_1]_s - [D_c])([D_2]_s - [D_c]) \qquad (i)$$

where $[D_1]_s$ and $[D_2]_s$ are the molarities of the dyes in the mixtures and $[D_c]$ is the molarity of the complex produced. If the fraction of dye 1 (Sky Blue) in the form of complex is expressed as a, then—

$$K = \frac{1-a}{a}\left(\frac{[D_2]_s}{[D_1]_s} - a\right)[D_1]_s \qquad (ii)$$

Then, by estimating values of a from the spectral changes produced at long wavelengths in admixture (Fig. 4), a value of K may be obtained. The spectra at 60°C. are consistent with a value of the order of $K_{60°} \sim 4 \times 10^{-6}$ M. when concentrations are expressed as molarities. The corresponding figure at 90°C. may be deduced from this by use of the heat of reaction of −15 kcal./mole, obtained from calorimetic measurements (see later). This leads to $K_{90} \sim 2{\cdot}5 \times 10^{-5}$ M. That a reaction constant of this order of magnitude leads to significant amounts of dye present in solution as complex at 90°C. is shown in Table I.

TABLE I

Total Concn. of Dye 1 (mM.)	Total Concn. of Dye 2 (mM.)	Calculated Fraction a of Dye 1 present as 1:1 Complex
0·02	0·02	0·35
0·02	0·05	0·60
0·02	0·20	0·88
0·05	0·05	0·50
0·05	0·10	0·72
0·05	0·20	0·86

Thus it might be expected that the uptake of these dyes from binary mixtures would differ considerably from that of the dyes applied singly. Furthermore, the greater reduction in adsorption will occur for that dye present in the mixture at the lower concentration.

II—ABSORPTION SPECTRA OF DYED CELLOPHANE

The spectra of the single dyes on Cellophane sheet showed marked departures from Beer's law at higher concentrations of dye. Consequently, interpretation of the results at these concentrations was difficult, and the spectra plotted in Fig. 5 all relate to lower values of dye adsorbed, at which single-dye spectra show a linear relation between optical density and dye adsorption.

The curve of Chlorazol Sky Blue FF alone on Cellophane is very similar to that deduced for the 1:1 complex in aqueous solution at room temperature, as may be seen by comparison with Fig. 2. This shows that the spectrum of Chlorazol Sky Blue FF is changed in the same way by combination either with cellulose or with a single Chrysophenine G molecule in solution. The spectral shifts produced by dyeing from mixture dyebaths indicate that the dye is partly adsorbed in the form of the complex, and the type of shift produced is consistent with this view. The curves all pass through a single isosbestic point, so that only single dye molecules and 1:1 complex molecules are adsorbed. This agrees with the deduction that higher complexes are not formed appreciably in solution at higher temperatures, and therefore will not be adsorbed at dyeing temperatures. Curves (ii)–(iv) all relate to dyeings from mixtures of the same ratio of blue to yellow dye, but with increasing salt concentration. Comparison of the three curves shows that the proportion of complex adsorbed increases with salt concentration. This is to be expected, as the complex will carry a charge of six units per ion and will consequently be more salt-sensitive than either of the single dyes. Curve (v) is that of a dyeing from a bath containing a

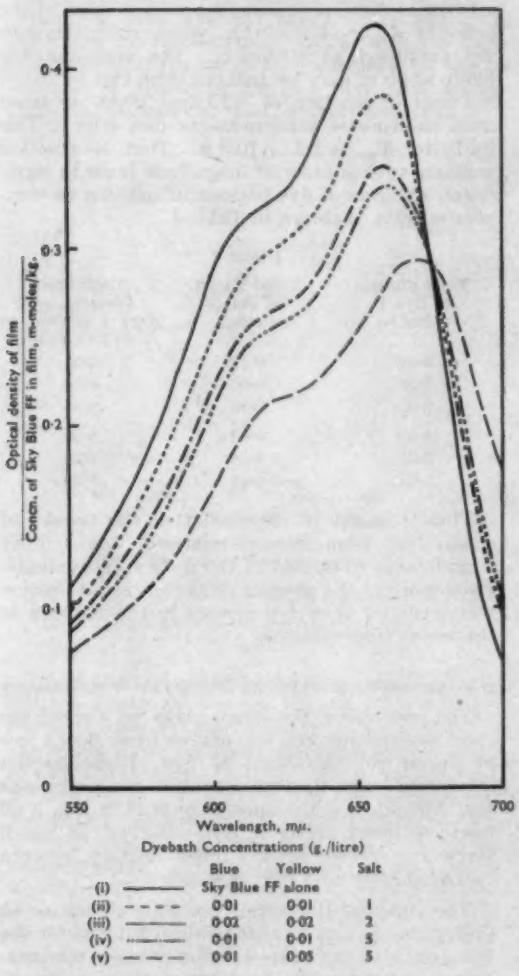


FIG. 5—Spectra of Dyed Cellophane

large excess of the yellow dye. Under these conditions there will be a high ratio of complex to blue dye in the bath, and a consequently high proportion of blue dye will be adsorbed in the form of complex.

It should also be noted that the spectral shifts produced by adsorption of an increasing proportion of complex parallel those produced in solution at room temperature by an increasing ratio of yellow to blue dye (Fig. 2). This, together with the similarity of the spectrum of Sky Blue on Cellophane to that of the 1:1 complex in solution, will be considered in greater detail on p. 276.

III—CALORIMETRIC RESULTS

The calorimetric measurements were made to determine the magnitude of the heat of interaction and also to confirm, if possible, the interpretation of the spectra obtained with solutions at room temperature. The heat changes produced on mixing solutions of the single dyes in varying proportions were measured, and the necessary heats of dilution of the dyes also determined. The results are summarised in Table II.

These figures agree with the interpretation of the spectra, for while the heats of reaction in the two experiments with excess of blue dye are nearly equal, that with excess of yellow dye produces a heat change about half as large again. This difference must be attributed to the formation of a considerable proportion of complexes richer in Chrysophenine G in the presence of an excess of this dye. On the assumption that in the first two mixtures virtually complete formation of 1:1 complex takes place, the heat of formation of the complex is −15·0 kcal./mole.

IV—EQUILIBRIUM ADSORPTION ON COTTON AT 90°C.

The results obtained by Neale and Stringfellow[3] give the impression that in a mixture the adsorption of Chlorazol Sky Blue FF is reduced to a greater extent than that of Chrysophenine G. This appears contrary to the hypothesis of the formation of a 1:1 complex in solution, until it is realised that their results refer in every case to adsorption from a bath containing an excess of the yellow dye, when expressed on a molar concentration basis. Consequently, further adsorption measurements were made to include dyeings from baths containing excess of blue dye. The first series was made from baths containing 5 g. sodium chloride per litre. Dyeings were made with each component singly and with mixtures containing various proportions of the two dyes. The results are given in Tables III–V. Each dyeing tabulated is the mean of two determinations made from identical starting conditions.

The interpretation of these results is rendered difficult by the lack of a completely adequate theoretical mechanism for adsorption of a single dye. If the Donnan theory is applied to these single dye data or to those of Neale and Stringfellow[3], a constant affinity value is not obtained.

TABLE II

Final Concn. of Chlorazol Sky Blue FF (mM.)	Final Concn. of Chrysophenine G (mM.)	Heat produced after Correction for Heat of Dilution (±0·5 cal.)	Ratio of Chlorazol Sky Blue FF to Chrysophenine G	Heat of Reaction per Mole of Lesser Constituent (kcal./mole)
1·91	1·00	14·6	1·91	−14·6 ± 0·5
2·03	0·50	7·9	4·06	−15·8 ± 1·0
0·50	1·00	11·4	0·50	−22·8 ± 1·0

Consequently, the theory cannot be used to give a complete check on the hypothesis of formation of a 1:1 complex of the two dyes in solution.

TABLE III

Adsorption of Chlorazol Sky Blue FF Alone

(5 g. NaCl per litre)

Dye in Soln. $[D]_s$ (mM.)	Dye on Fibre $[D]_f$ (m-mole/kg.)
0·035	3·46
0·084	6·35
0·176	7·94
0·245	8·70

TABLE IV

Adsorption of Chrysophenine G Alone

(5 g. NaCl per litre)

Dye in Soln. $[D]_s$ (mM.)	Dye on Fibre $[D]_f$ (m-mole/kg.)
0·063	1·94
0·132	3·27
0·295	5·46

Nevertheless, the results show at least a qualitative agreement with the hypothesis. It is apparent from the mass action equation (i) for formation of such a complex that the concentration of unassociated dye relative to total concentration of that dye will be less for the component that is present at the lower total concentration. Consequently, the adsorption of that dye should suffer the greater reduction. It is also apparent that the larger the excess of the second dye, the larger will be the reduction in the adsorption of the first. Furthermore, the symmetry of the mass action equation should lead to similar behaviour for either dye. These considerations are based on the assumption that the substantivity of the complex is much less than that of either constituent, which would be expected from its high charge and is confirmed by the chromatography results already described. A complete correlation on this oversimplified model can hardly be expected, since the effect on the fibre potential of the adsorption of two dyes together has been ignored.

This approximate description of the system may be checked by calculating the fraction representing the ratio of the relative adsorption of the two dyes—

$$\frac{[D_1]_f \text{ in mixture}}{[D_1]_f \text{ alone}} \times \frac{[D_2]_f \text{ alone}}{[D_2]_f \text{ in mixture}} = y$$

and plotting it against—

$$\frac{[D_1]_s}{[D_2]_s} = R$$

the ratio of the lesser to the greater constituent of the mixture, for cases where $[D_2]_s > [D_1]_s$. For cases where $[D_2]_s < [D_1]_s$ the reciprocals of both fractions have been calculated and similarly plotted. Values of $[D_1]_{f\ \text{alone}}$ and $[D_2]_{f\ \text{alone}}$ were read off from graphs of the single dye data at the appropriate values of $[D_1]_s$ and $[D_2]_s$.

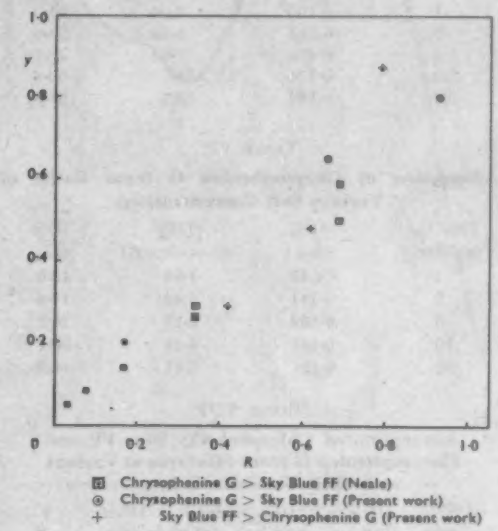


FIG. 6

The results are shown in Fig. 6, both from this research and from the previous work of Neale and Stringfellow[2]. In view of the oversimplification of the system, the correlation is reasonably good, and there is no significant difference between the

TABLE V

Adsorption of Chlorazol Sky Blue FF and Chrysophenine G from Mixtures

(5 g. NaCl per litre)

Chlorazol Sky Blue FF in Soln. $[D_1]_s$ (mM.)	Chrysophenine G in Soln. $[D_2]_s$ (mM.)	Chlorazol Sky Blue FF on Fibre $[D_1]_f$ (m-mole/kg.)	Chrysophenine G on Fibre $[D_2]_f$ (m-mole/kg.)
0·043	0·066	1·84	1·34
0·047	0·274	0·56	3·30
0·067	0·072	3·55	1·66
0·089	0·070	4·49	1·33
0·185	0·115	5·91	1·05
0·265	0·112	7·02	0·67

behaviour with either component in excess in the mixture. The present results also agree well on this basis with those obtained previously, in spite of considerable differences in single dye adsorption isotherms due to use of different fibre samples.

Neale and Stringfellow[2] also give a series of measurements on the effect of salt concentration on adsorption from a mixture of constant constitution, again with Chrysophenine G in excess. A similar series has been measured in this work with Chlorazol Sky Blue FF in excess. The results are given in Tables VI to VIII.

TABLE VI

Adsorption of Chlorazol Sky Blue FF from Baths of Various Salt Concentrations

$[NaCl]_s$ (g./litre)	$[D]_s$ (mM.)	$[D]_f$ (m-mole/kg.)	$\frac{[D]_f}{[D]_s}$
1	0·195	2·96	15·2
2	0·191	4·58	24·0
5	0·176	7·94	45·1
10	0·176	10·7	60·8
20	0·166	15·9	95·8

TABLE VII

Adsorption of Chrysophenine G from Baths of Various Salt Concentrations

$[NaCl]_s$ (g./litre)	$[D]_s$ (mM.)	$[D]_f$ (m-mole/kg.)	$\frac{[D]_f}{[D]_s}$
1	0·143	1·95	13·6
2	0·141	2·42	17·2
5	0·132	3·27	24·8
10	0·141	4·25	30·1
20	0·131	5·87	44·8

TABLE VIII

Adsorption of Chlorazol Sky Blue FF and Chrysophenine G from Mixtures at Various Salt Concentrations

[NaCl] (g./litre)	$[D_1]_s$ (mM.)	$[D_2]_s$ (mM.)	$[D_1]_f$ (m-mole/kg.)	$[D_2]_f$	$\frac{y'}{R}$
1	0·195	0·138	2·14	0·75	0·77
2	0·188	0·136	3·26	0·98	0·81
5	0·185	0·115	5·91	1·05	0·84
10	0·182	0·134	7·72	2·12	1·02
20	0·170	0·132	10·8	3·25	1·07

These results may be treated in a similar way to those at constant salt concentration. To allow for the small variations in dyebath concentration the ratio y' has been calculated as—

$$y' = \frac{\left(\frac{[D_2]_f}{[D_2]_s}\right)_{\text{in mixture}}}{\left(\frac{[D_2]_f}{[D_2]_s}\right)_{\text{alone}}} \times \frac{\left(\frac{[D_1]_f}{[D_1]_s}\right)_{\text{alone}}}{\left(\frac{[D_1]_f}{[D_1]_s}\right)_{\text{in mixture}}}$$

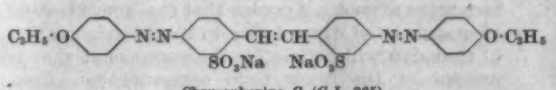
and R as before—

$$R = \frac{[D_2]_s}{[D_1]_s}$$

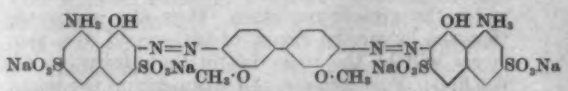
Finally, the ratio y'/R has been calculated for each salt concentration and is given in the last column of Table VIII. It is seen that this ratio increases with increasing salt concentration. This must be attributed to a greater increase of substantivity of the complex compared with that of the single dyes as the salt concentration increases, as would be expected from its higher charge. The results indicate that this effect outweighs any increase in complex formation due to increasing ionic strength of the solution. This latter is probably fairly small for this pair of dyes, whose mechanism of complexing is considered in greater detail in the Discussion section. Unfortunately, the data of Neale and Stringfellow[2] cannot be calculated on the same basis, since these authors publish no results for the adsorption of Chrysophenine G alone at varying salt concentration.

Discussion

The conclusions to be drawn from each type of experiment have already been considered in the Results section. The general significance of these results may now be discussed. There can be little doubt that 1:1 complex formation in solution persists up to 90°C. and modifies dyeing behaviour in mixtures. It is also apparent that this occurs to a much greater extent than the corresponding aggregation of a single dye. This is probably due to the structure of the two dyes, for while Chlorazol Sky Blue FF has its charged groups at the ends of the linear molecule, the corresponding groups of Chrysophenine G are near the centre—



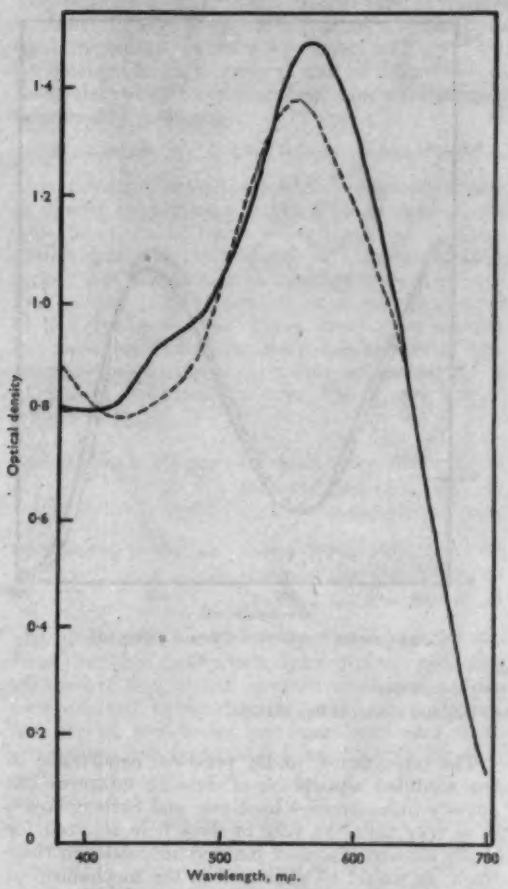
Chrysophenine G (*C.I.* 365)



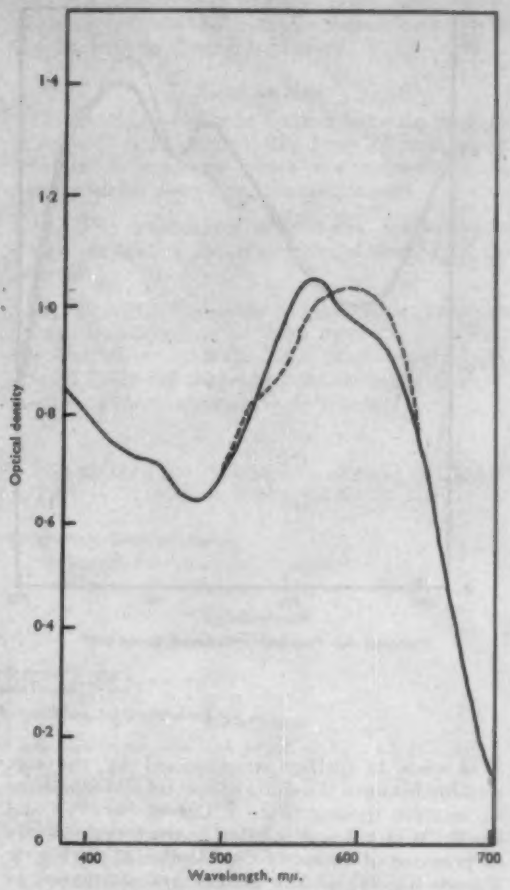
Chlorazol Sky Blue FF (*C.I.* 515)

Thus the formation of a complex by superposition of the two linear and planar molecules entails no very close approach of charged centres. This becomes important only on the addition of a second molecule of either species, and the results indicate that the tetrabasic Chlorazol Sky Blue FF does not form appreciable amounts of higher complexes even at room temperature, while the dibasic Chrysophenine G does so to a limited extent at the lower temperature but not at 60°C. On the other hand, self-aggregation of either dye by similar superposition must involve very close approach of the identically disposed charge groups, and will consequently be much less favourable energetically.

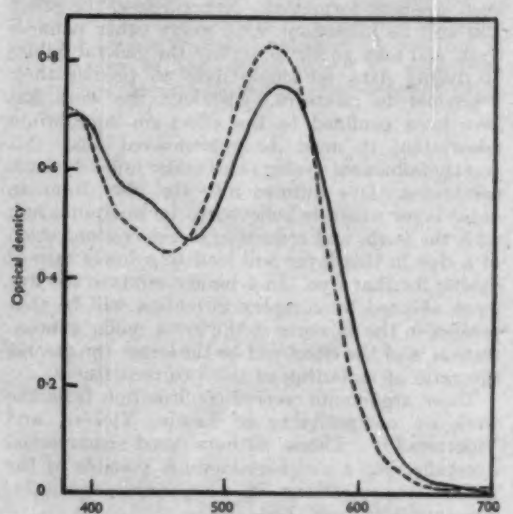
This work also provides considerable evidence that the mechanism of complex formation is similar to that of dyeing. The close similarity between the spectra of Chlorazol Sky Blue FF adsorbed on Cellophane and of the 1 : 1 complex in solution has already been indicated. The shifts in the spectrum of Chrysophenine G on adsorption and on complex formation are also similar. This indicates that the same forces operate in the formation of dye–substrate and dye–dye bonds.



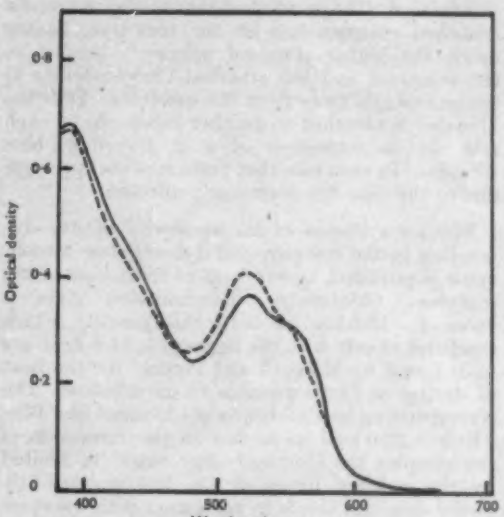
Chlorazol Fast Helio 2RKS + Chlorazol Dark Green PLS



Chlorazol Fast Pink BKS + Chlorazol Dark Green PLS



Chlorazol Fast Helio 2RKS + Chlorazol Yellow 6GS



Chlorazol Fast Pink BKS + Chlorazol Yellow 6GS

(FIG. 7)

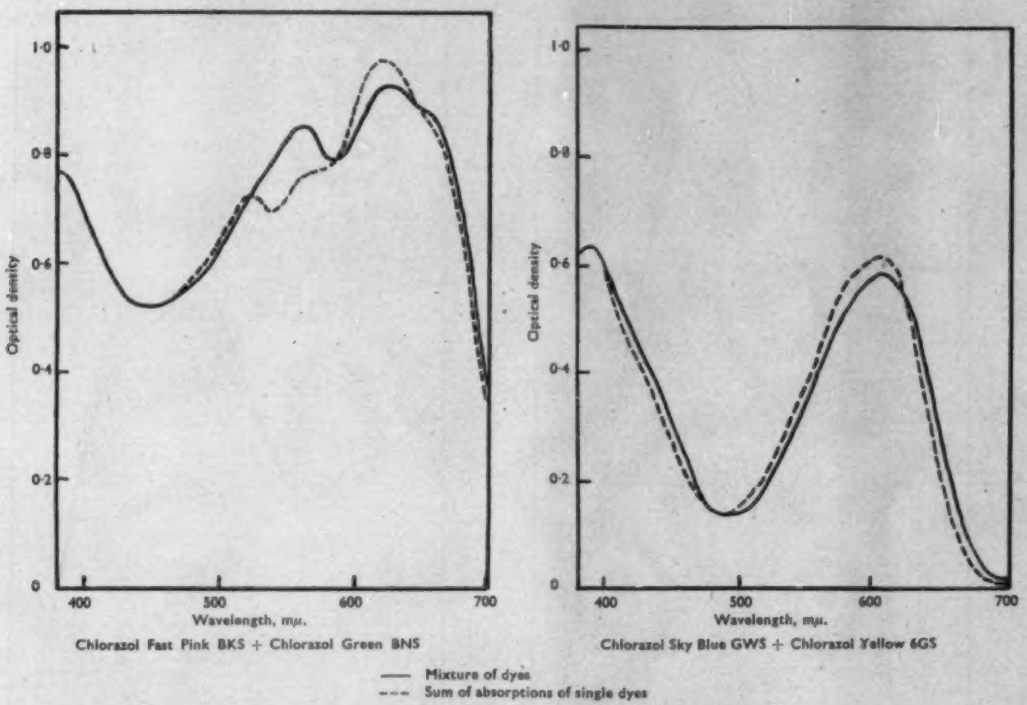


FIG. 7— Deviations from Additivity of the Absorption Spectra of Dye Mixtures

This view is further strengthened by the correlation between the shifts of spectra on Cellophane in mixture dyeing (Fig. 5, Curves (ii)–(v)) and the shifts in aqueous solution at room temperature in presence of excess of Chrysophenine G (Fig. 3, Curves (iii)–(v)). The former are attributed to adsorption of some of the blue dye as complex, and the latter to formation of complexes richer in yellow dye. The most probable configuration for adsorbed complex is with the blue dye, having much "the higher standard affinity"[7], bonded to the substrate and the attached Chrysophenine G ion on the side away from the substrate. Thus the blue dye is attached to another substance on each side, as in formation of a 2 : 1 yellow : blue complex. In each case that portion of the spectrum due to the blue dye is similarly affected.

Further evidence of the similarity of dye–dye bonding in the complex and dye–cellulose attachment is provided by the heat of formation of the complex. Calorimetric determination gives a value of —15·0 kcal./mole for this quantity, which compares closely with the figure of —14·0 kcal. per mole found by Marshall and Peters[7] for the heat of dyeing of Chrysophenine G on cellulose. The corresponding heat of dyeing of Chlorazol Sky Blue FF is —22·0 kcal./mole, but in the formation of the complex the binding energy would be limited by the valency forces of the less energetically bound dye. All this is in agreement with views on the importance of non-polar van der Waals forces in dyeing phenomena, which are considered in detail elsewhere[8, 9].

The importance, under practical conditions, of the modified adsorption of dyes in mixtures has already been stressed by Neale and Stringfellow[2]. It is true that the pair of dyes here selected for study showed the most marked anomalies in their work, as would be expected on the mechanism of complex formation outlined above, since the charge distributions of the two dyes are ideally suited to such complex formation. Nevertheless, the effect will still be important with many other pairs of dyes, and may go far to explain the general failure of dyeing data on single dyes to predict their behaviour in mixtures. Although the work has here been confined to the effect on equilibrium adsorption, it must be remembered that this greatly influences dyeing rates under finite dyebath conditions. Dye diffuses into the fibre from an outer layer which is believed to be in equilibrium with the bath, and reduction of the concentration of a dye in this layer will lead to a lower rate of dyeing for that dye. In a binary mixture the dye more affected by complex formation will be that present in the mixture at the lower molar concentration, and the effect will be the larger the greater the ratio of molarities of the two constituents.

These arguments receive confirmation from the work on compatibility of Lemin, Vickers, and Vickerstaff[1]. These authors used commercial dyestuffs, but a rough estimate is possible of the molar concentrations in the initial dyebaths. The molarity will be—

$$\text{M.} = \frac{\text{Depth (\%)} \times \text{Purity (\%)}}{\text{Liquor ratio} \times \text{Mol. wt.} \times 10^4}$$

The percentage purity is equal to (100 — diluent content %) and may be estimated for the dyes used in binary mixture by these authors. The percentage depth is known, and if differences in molecular weight are neglected the approximate relationship becomes—

$$M. \propto \text{Depth}\ (\%) \times (100 - \text{Diluent content}\ (\%))$$

The above authors attempted to predict the course of dyeing from binary mixtures from their single dye data. As a result of comparison of these predictions with the results of dip tests, they picked out three dyes as tending always to dye first in spite of unfavourable exhaustion and time of half-dyeing values. Three other dyes showed the converse behaviour very markedly. If the quantity proportional to molar concentration is calculated for these six dyes, the figures are as follows—

Dyes tending to dye first:	Chlorazol Yellow 6G	85
	Chlorazol Dark Green PL	68
	Chlorazol Green BN	75
Dyes tending to dye last:	Chlorazol Fast Helio 2RK	15
	Chlorazol Fast Pink BK	18
	Chlorazol Sky Blue GW	10

The mean value for all the dyes used is about 40. Thus the three dyes which dye first from mixtures are used at high molar concentrations and will be least affected by complexing, while those dyeing last are at low molar concentrations and their adsorption will be retarded in mixture by this effect.

The most pronounced departures from expected behaviour were obtained with combinations of these dyes. Absorption spectra of a number of these combinations were measured at room temperature, using arbitrary concentrations of commercial dyes. The spectra are plotted in Fig. 7, in which the full line is the mixture spectrum and the broken line that obtained by addition of the spectra of the single dyes. In each case departures from additivity are observed, indicating complex formation in mixture. This supports the above hypothesis that complex formation explains the anomalous dyeing behaviour.

Conclusions

(1) A 1:1 complex is formed between Chrysophenine G and Chlorazol Sky Blue FF in aqueous solution in amounts which are appreciable at dyeing temperatures and concentrations.

(2) The mechanism of complex formation is similar to that of the adsorption of these dyes on cellulose.

(3) Complex formation in solution explains the change in adsorption of these dyes when applied from mixture dyebaths and may explain the general failure of data obtained on single dyes in predicting their behaviour in mixtures.

* * *

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Package Dyeing

II—The Effect of Liquor Ratio on Direct Dyeing

W. ARMFIELD, J. BOULTON, and J. CRANK

Package dyeing has been carried out on a laboratory apparatus with a number of dyes, each in 6 and 20 volumes, with enough sodium chloride added at the commencement of dyeing to give roughly the same final exhaustion at the two liquor ratios for any particular dye. After 30 min., with the same rate of flow, the 6-vol. dyeing was always more level than the corresponding 20-vol. dyeing.

Special consideration is given to a class C dye which without added salt has a very high exhaustion in low volumes, due to the salt contained in the dye. Here also the 6-vol. dyeing was more level than the 20-vol. dyeing after 30 min., although the former package was more than twice as heavily dyed. These observations have been confirmed by dyeing commercial viscose rayon cakes on a laboratory cake-dyeing machine with single dyes and mixtures.

The advantage gained by working in low volumes is shown to be due to an increase in the dyebath cycle rate, and an expression is given showing the relationship of this to yarn weight and rate of flow. The variation in yarn weight in bulk cake-dyeing is considered in so far as it affects liquor ratio and dyebath cycle rate.

In the second part of the paper the results are examined in the light of a theoretical model of package dyeing previously described. It is found that, when certain parameters are chosen to give a fit with measured skein-dyeing curves, the main conclusions about package dyeing based on the experimental results are substantiated by the model. At the same time, more information is provided on the rate of exchange of dyes between liquor and yarn within the package, and on the influence of the three basic parameters on which package-dyeing behaviour depends.

Introduction

This paper has a twofold interest. Firstly, it is concerned with the very practical problem of reducing the time of dyeing, and special consideration is given to the effect of liquor ratio. Secondly, it is of theoretical interest in providing more information about the processes of exchange and migration of dye which take place within the package. The form of presentation of the paper reflects these two interests.

In a previous paper [1] the effect of the rate of flow of dye liquor through a package was studied, and since then Boulton and Crank [2] have worked out a theoretical model for package dyeing. One factor, however, which has not so far received special consideration is the effect of liquor ratio, which is a frequent variable in all dyehouses. In the Droylsden dyehouse, for example, cake dyeing is carried out in liquor ratios varying between 11 : 1 and 27 : 1. Two obvious advantages in dyeing in as low a volume of liquor as possible are economy in time and steam and, to a lesser degree, in dyes, chemicals, and auxiliary products. Experiments and experience have shown that it is advantageous to work to dyebath exhaustions of not less than about 75%, so that if two batches in different volumes are to be dyed to the same depth of colour using the same percentage of dye, the colour can be matched by using different salt concentrations to compensate for the volume differences. Experiments were therefore carried out in 6 vol. and 20 vol. on a number of single dyes using the same weights of yarn and dye and the same rate of flow, but with different salt concentrations, so that the final exhaustions were the same for a particular dye. It was considered advisable to work to the lowest practicable liquor ratio, which preliminary trials showed to be 6 : 1.

Package Dyeing

EXPERIMENTAL

The apparatus described previously [1] was not suitable for such small-volume dyeings, because the temperature of the dyebath could not be kept sufficiently constant. Furthermore, the small excess of liquor was not sufficient to enable the rate of flow to be measured as described. A special apparatus was designed (Fig. 1) which overcame those difficulties and allowed dyeings to be made in 6 and 20 vol.

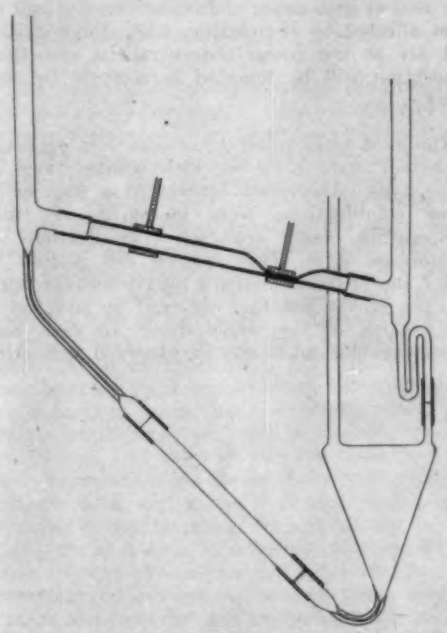

FIG. 1

The apparatus consists of a 100-c.c. dye vessel with extended air inlet at the top. The bottom of the vessel is connected to the yarn tube, which is 182 mm. long by 9 mm. internal diameter. The yarn tube is connected at the other end to a capillary tube (1·5 mm. diameter), which is attached to the left-hand upright limb of the apparatus.

The latter is connected by rubber tubing to the right-hand upright limb, which in turn is connected to the dye vessel. This rubber tubing passes under two plungers, as shown in Fig. 2. The plungers are depressed by cams driven by a Klaxon geared motor working at 17 r.p.m., in such a way that during the time that either plunger is depressed to close the tube completely the other plunger allows the tube beneath it to remain open. With the motor switched on, vacuum is applied to the left-hand upright limb, sucking the dye liquor through the yarn, and then allowing it to pass through the rubber tubing. The rate of flow is adjusted by controlling the degree of vacuum, and is measured by observing the time taken for the siphon tube (capacity 3·2 c.c.) on the right-hand upright limb to fill. The apparatus is immersed in a thermostatically controlled water bath arranged so that the level of the water leaves the top of the siphon tube showing.

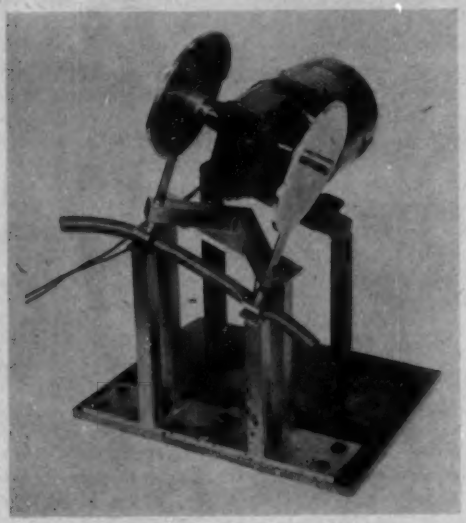

FIG. 2

The yarn tube is packed by drawing through it a skein of 10 g. oven-dry weight. One end of the skein is drawn flush with one end of the tube, and is held in position by passing through it a thin piece of wire, which is bent back along the walls of the tube for about ¼ in. The skein is now cut flush with the other end of the tube, and by weighing the tube before and after packing the weight of the yarn is ascertained. The yarn used in these experiments was 150/27 viscose rayon, pretreated three times with distilled water at 60°C. for 10 min., squeezed, dried, and conditioned.

EXPERIMENTAL RESULTS

The dyes listed in Table I were applied in 6 and 20 vol. at 95°C. for 30 min. at a rate of flow of 2 c.c./min. per gram of yarn. The dyeings in 20 vol. contained 5% sodium chloride from the commencement of dyeing, but the dyeings in 6 vol. contained only 0·6% sodium chloride, so as to

TABLE I

Depth	Dye	Makers	Exhaustion (%) 20 vol. 5% NaCl	Exhaustion (%) 6 vol. 0·6% NaCl	Relative Levelness in 6 vol. and 20 vol.*
0·5%	Solophenyl Blue 4RL 200	Gy	81·7	88·9	+
0·44%	Solophenyl Navy Blue FGL 225	Gy	65·8	64·5	+
0·66%	Chlorazol Dark Blue B 150	ICI	84·5	87·7	+
0·5%	Durazol Blue G 200	ICI	80·1	86·3	+
0·5%	Durazol Blue 2R 200	ICI	84·2	84·9	++
1·0%	Durazol Blue 4GS	ICI	53·0	57·0	+
0·5%	Durazol Blue 2GN 200	ICI	78·2	82·5	+
0·5%	Chlorantine Fast Blue GLL 200	CDC	86·4	92·7	+
0·4%	Chlorantine Fast Blue 3GLL 250	CDC	59·0	52·8	+
0·33%	Diphenyl Brilliant Blue FF 300	Gy	88·3	90·6	++
0·33%	Solar Blue F New 300	S	40·0	40·9	+
0·5%	Durazol Blue 3R 200	ICI	80·9	82·0	+
0·67%	Durazol Grey VG 150	ICI	70·2	67·6	+
0·63%	Durazol Orange 2G 160	ICI	55·5	61·6	+
0·66%	Durazol Orange 4R 150	ICI	58·8	60·9	+
0·33%	Solar Discharge Orange GL 300	S	75·0	76·1	+
0·59%	Solar Orange RGL 170	S	59·5	57·1	+
0·56%	Solar Yellow 2R 180	S	77·2	74·6	++
1·0%	Solophenyl Brown GL	Gy	57·4	59·5	++
1·0%	Solophenyl Brown RL	Gy	61·0	64·8	+
1·0%	Chlorantine Fast Brown 8RLL	CDC	75·0	82·6	++
0·76%	Benzanil Fast Brown 5RL 132	YDC	58·5	65·5	+
0·66%	Solar Brilliant Red BA 150	S	42·0	44·0	+
0·66%	Durazol Scarlet 2G 150	ICI	60·9	61·7	+
0·59%	Solophenyl Bronze B 170	Gy	55·5	63·2	+
0·55%	Sirius Supra Green BTL 182	FBy	62·8	62·9	+
0·60%	Sirius Supra Olive GL 167	FBy	86·5	89·5	+

* + 6-vol. more level than 20-vol. dyeing ++ 6-vol. much more level than 20-vol. dyeing

give approximately equal final dyebath exhaustions for any particular dye. In every case, the 6-vol. dyeing was more level than the corresponding 20-vol. dyeing, the levelness being assessed visually by the difference in depth of colour between the inflow and outflow ends of the packages.

For convenience in these experiments, the added electrolyte was present from the start, but in practice the dye is salted on gradually, and so it was considered necessary to determine the effect of volume on levelness under conditions corresponding more nearly to works practice. Under these conditions, where dyeing is commenced without added electrolyte, the effect of the electrolyte in the dye itself is to cause a higher rate of dyeing and greater equilibrium exhaustion the lower the dyebath volume.

Experiments were carried out with Chlorazol Brown M 145 in 6 and 20 vol. at 95°C. and 2 c.c. per min. per gram of yarn for 3, 5, 30, and 60 min. with no added electrolyte. The exhaustion and the levelness were determined by the following procedure and are shown diagrammatically in Fig. 3 and 4. A number of 0·5-g. portions (oven-dry weight) were cut from each end of the package, and the dye was removed by boiling in a 15% solution of pure pyridine under reflux. The optical density of each solution was measured on an E.E.L. colorimeter, and by expressing the optical density corresponding to the outflow end of the package as a decimal fraction of that of the inflow end a levelness factor (L.F.) was obtained. Thus the nearer the levelness factor is to unity, the greater is the levelness of the package. In Fig. 3 and 4 the large squares represent the whole of the package dyed uniformly to 100% dyebath exhaustion (i.e. 100 dye units). Thus the shaded portions under each line represent the distribution of the dye on the package.

GENERAL DISCUSSION OF RESULTS

The results in Table I show that, in principle, for a given final exhaustion, dyeing in as low a volume as possible is advantageous. Fig. 3 and 4, referring to dyeings with no added salt, show that initially a higher rate of dyeing and a higher degree of exhaustion are obtained in low volumes than in high volumes, primarily because of electrolyte concentration resulting directly from the salt contained in the dye. The initial levelness in low volumes is not as good as in high volumes, but rapidly improves and in this particular case becomes better after only 30 min. dyeing. After this time of dyeing, the dyebath exhaustion is twice as large with the lower volume, and the package is more evenly dyed from end to end. In order to obtain the same depth of colour on both packages, the higher-volume dyeing would require salt additions, but during this salting period the lower-volume dyeing would continue to become more level owing to dye migration. Thus the overall picture is very much in favour of the low-volume dyeing.

In order to account for the above behaviour when no salt is added, consideration should be given to the exhaustion of the dyebath as a whole and also to the take-up of dye as the liquor circulates through the yarn. With regard to the first, although the salt : dye ratios are the same in both volumes, the respective salt and dye concentrations are in the inverse ratio of the dyebath volumes. Thus the lower-volume liquor with its higher salt concentration has a higher rate of dyeing and also a higher equilibrium exhaustion. This is a well known fact and is illustrated in Fig. 5 and 6, which show skein-dyeing curves in 12 and 20 vol. respectively, there being no salt added in either case, though salt is contained in the dye. In package dyeing the whole mass of yarn is in contact with

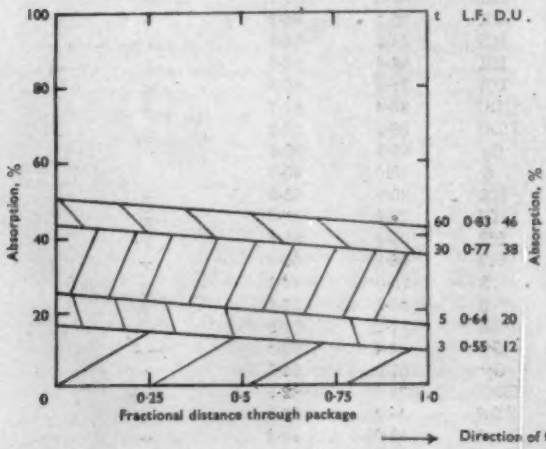

FIG. 3— 20-vol. Liquor

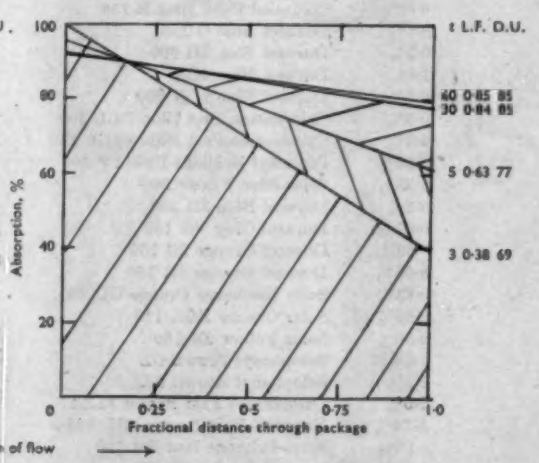

FIG. 4— 6-vol. Liquor

t = Time of dyeing (min.)
L.F. = Levelness factor
D.U. = Dye units (the dyebath contains initially 100 D.U.)

FIG. 3 and 4— Experimental Package-dyeing Curves
(0·60% Chlorazol Brown M 145)

the dye only when the dye liquor is circulating through the package. Therefore, since the dyebath cycle rate is greater in the case of the lower volume in the ratio of the two volumes, this means a further increase in dye take-up in addition to the effect of salt concentration, because the yarn is in contact with more dye in a given time.

Considering now the liquor passing through the package as a series of successive thin liquid layers, the first layer is more highly exhausted in the low-volume dyeing for any given distance of penetration through the package, because of the higher salt concentration. Each successive layer behaves similarly, until equilibrium is reached between dye adsorbed on the inflow end of the package and the dye in the liquor passing through. When this stage is reached, dyeing takes place only further along the package. Fig. 4 (6 vol.) shows quite clearly that after 3 min. the inflow end of the package has taken up slightly more dye than it will actually contain at equilibrium. Hence no further dyeing can take place at this point, but the dye take-up along the length of the package increases to satisfy equilibrium requirements. Fig. 3 (20 vol.) shows that after 3 min. the inflow end of the package is far from having reached equilibrium exhaustion, and so dye continues to be taken up along the whole package length. Thus in the early stages of dyeing the lower-volume dyeing is more uneven along the length of the package, and the amount of dye absorbed is greater, but on continued liquor circulation the levelness of the low-volume package increases at a greater rate. This is mainly due to the higher dyebath cycle rate, which is partly dependent upon liquor:yarn ratio—

$$\text{Time required for one dyebath cycle} = \frac{\text{Liquor ratio} \times \text{Yarn weight}}{\text{Rate of flow}}$$

Cake Dyeing

In the foregoing experiments the yarn weight and the rate of flow have been kept constant, but the volume of liquor, and hence the liquor ratio, have been altered. Considering now a cake-dyeing machine in which the liquor is circulated by means of a centrifugal pump, a different set of conditions exists. On such a machine with a full load of cakes there is the maximum rate of liquor circulation. By decreasing the number of cakes, a greater resistance is offered to the flow of liquor because of the smaller number of capillary air spaces, so that the rate of flow of liquor is correspondingly decreased. If the same volume of liquor were used as for the full load, the dyebath cycle rate would also be less, but if the liquor ratio were kept the same, the dyebath cycle rate also would remain the same. It follows, therefore, that for any one machine the best results should be obtained by using the lowest possible volume of liquor.

It should be understood that, in cake dyeing, all the liquor which passes through the pump does not pass through the mass of yarn. Much of the liquor circulates via leakages between cakes and cake holders, and in fact through any joining which is not a perfect seal. It is only the liquor which passes through the mass of yarn, therefore, which represents the effective flow rate.

The expression showing the relationship between dyebath cycle rate, liquor ratio, yarn weight, and rate of flow is perfectly valid, but for any one cake-dyeing machine, as explained above, the ratio of the yarn weight to the effective rate of flow must be approximately invariable.

In Table II, columns A and B represent the conditions for different liquor ratios used in the above experiments. Assuming that column A represents a full load under bulk dyeing conditions in a liquor ratio of 20:1, then column C indicates the conditions for a half-load in the same total volume of liquor, and column D a half-load in half the total volume of A.

TABLE II

	A	B	C	D
Yarn weight, g.	10	10	5	5
Liquor ratio	20	6	40	20
Total volume, c.c.	200	60	200	100
Rate of flow, c.c./min.	10	10	5	5
Period of dyebath cycle, min.	20	6	40	20

In order to study the effect of liquor ratio experimentally, but under conditions similar to bulk practice, a cake-dyeing machine was constructed such that one commercial-size cake could be dyed in 6–20 vol., or three cakes in 6 vol.

The dye chamber of this machine consists of a cylindrical stainless-steel tank, 7 in. in diameter × 16 in. high, surrounded by a water jacket, 10 in. in diameter × 15 in. high. The latter contains a circular 1500-watt heating element coupled to a Sunvic TS2 regulator and relay. The dye chamber is connected to a centrifugal pump with an outflow capacity of approx. 2½ gal./min. The following experiments were carried out on this machine.

EXPERIMENTAL

Viscose rayon cakes 150/27 HT 40 were dyed with class B and class C direct dyes, salt additions being made where necessary, so that the final dyebath exhaustions were approximately the same in 6 and 20 vol. Where possible, the salt additions were controlled so as to give an even rate of dye exhaustion throughout the dyeing period. This control was obtained by determining the dyebath exhaustion during the actual course of dyeing.

Single cakes were dyed at 90°C. for 2 hr., the dye liquor being circulated from inside to outside for the whole of the dyeing time. The evenness was assessed by visual examination of yarn taken from the inside, middle, and outside of the cakes and knitted in consecutive panels. Before knitting, however, 12 g. of yarn was first removed from the outside of the cakes, because, with inside-to-outside circulation, the outer layers of yarn are often abnormally thin in depth.

TABLE III

Depth	Dye	Makers	Exhaustion (%) 20 vol.	Exhaustion (%) 6 vol.	Relative Levelness in 6 vol. and 20 vol.*
0·69%	Chlorazol Brown M 145	ICI	71	85	+
0·63%	Solar Yellow 2R 180	S	68	67	++
1·0%	Solar Orange RGL	S	48	49	+
0·33%	Solar Discharge Orange GL 300	S	76	70	+
1·0%	Solophenyl Brown RL	Gy	61	69	+
0·5%	Durazol Blue 2R 200	ICI	84	80	++
0·67%	Durazol Grey VG 150	ICI	86	86	++
2·0%	Coprantine Rubine RLL 300	CDC	76	80	++

* + 6-vol. more level than 20-vol. dyeing ++ 6-vol. much more level than 20-vol. dyeing

The results obtained with single dyes and with mixtures are shown in Tables III and IV respectively. With the dye mixtures it was impracticable to determine the exhaustion during the course of dyeing, so one set of salting conditions was adopted for all the dyeings in 6 vol., and another set of conditions for the dyeings in 20 vol. Both sets of conditions were based on observed averages for single dyes.

TABLE IV

	Dye Mixture	Makers	Relative levelness* in 6 vol. and 20 vol.†
0·29%	Solar Green 5GL 155	S	++
0·036%	Durazol Blue 2R 200	ICI	
0·48%	Sirius Supra Olive GL	FBy	
0·78%	Sirius Supra Olive GL	FBy	×
0·11%	Durazol Blue 2R 200	ICI	
0·32%	Solar Discharge Orange GL 300	S	×
0·053%	Solar Orange RGL 155	S	
0·47%	Chloramine Scarlet 4B 150	S	+
0·29%	Solar Red 2BL	S	
0·85%	Durazol Brown 8RS	ICI	+
0·068%	Solophenyl Bronze BL 170	Gy	
0·02%	Durazol Blue 2R 200	ICI	
0·26%	Durazol Blue 2R 200	ICI	++
0·13%	Solar Discharge Orange GL 300	S	
0·049%	Solophenyl Bronze BL 170	Gy	
0·44%	Solar Orange RGL 155	S	++
0·011%	Durazol Blue 2R 200	ICI	
0·29%	Durazol Brown 8RS	ICI	
0·17%	Solar Discharge Orange GL 300	S	++
0·30%	Solophenyl Brown GL	Gy	
0·11%	Solophenyl Bronze BL 170	Gy	

* × 6-vol. slightly more level than 20-vol. dyeing
\+ 6-vol. more level than 20-vol. dyeing
++ 6-vol. much more level than 20-vol. dyeing

† Method of salting—

20-VOL.

0·4% NaCl added at start
0·6% NaCl added after 20 min.
0·9% NaCl added after 40 min.
1·4% NaCl added after 60 min.
1·7% NaCl added after 80 min.

6-VOL.

0·1% NaCl added after 20 min.
0·1% NaCl added after 40 min.
0·1% NaCl added after 60 min.
0·15% NaCl added after 80 min.
0·15% NaCl added after 100 min.

Experiments were next carried out in which three cakes were dyed in 6 vol., using the same method of salting as stated in Table IV, except with Chlorazol Brown M, for which no salt was used. The results are given in Table V.

TABLE V

	Dyes	Remarks
0·69%	Chlorazol Brown M 145	Bottom and middle cakes a little more level than top cake, but none as good as 1 cake in 6 vol., and all better than 1 cake in 20 vol.
0·5%	Durazol Blue 2R 200	Middle cake more level than top and bottom cakes, but not quite as good as 1 cake in 6 vol., and all better than 1 cake in 20 vol.
0·67%	Durazol Grey VG 150	Middle cake better than bottom, bottom better than top. Better than 1 cake in 6 vol., and all much better than 1 cake in 20 vol.
	MIXTURE	
0·29%	Solar Green 5GL 155	Bottom and middle cakes better than top and better than 1 cake in 6 vol., and all much better than 1 cake in 20 vol.
0·036%	Durazol Blue 2R 200	
0·48%	Sirius Supra Olive GL	

In order to see whether the disadvantage of dyeing in 20 vol. compared with 6 vol. could be offset by increasing proportionately the rate of flow of dye liquor, experiments were carried out on the laboratory package-dyeing apparatus described earlier (Fig. 1). Dyeings made in the presence of 5% sodium chloride in 20 vol. at 95°C. for 30 min. with a rate of flow of 6·66 c.c./min. per gram of yarn were compared with dyeings in 6 vol. made with 0·6% salt at 2 c.c./min. per gram of yarn, so that the cycle rate was the same. The results are shown in Table VI.

DISCUSSION OF RESULTS

Inspection of Tables III and IV shows that, both with single dyes and with mixtures, better levelness is obtained with one cake dyed in 6 vol. than in 20 vol. for approximately the same depth of colour. Also, Table V shows results in the order expected, i.e. 3 cakes in 6 vol. dye similarly to 1 cake in 6 vol. but better than 1 cake in 20 vol. Of the three cakes dyed together, the top cake of each lot was the most unevenly dyed from inside

to outside, owing to the increased pressure required to penetrate the top cake due to the greater head of liquor. In bulk practice this effect is negligible owing to the much higher pump pressures employed. Table VI shows that even in 20 vol. the levelness nearly approaches that obtained in 6 vol. provided that the dyebath cycle rate is the same.

TABLE VI

	Dye	Relative Levelness in 6 vol. and 20 vol.*
0·56%	Solar Yellow 2R 180	=
1·0%	Durazol Fast Blue 4 GS	×
0·5%	Durazol Blue 2R 200	×
0·63%	Durazol Orange 2G 160	×
0·67%	Durazol Grey VG 150	×
1·0%	Solophenyl Brown GL	×
0·55%	Sirius Supra Green BTL 187	×
1·0%	Solophenyl Brown RL	×
0·33%	Diphenyl Brilliant Blue FF 300	+

* = No difference in levelness
× 6-vol. slightly more level than 20-vol. dyeing
\+ 6-vol. more level than 20-vol. dyeing

These experiments confirm, therefore, that it should be advantageous in bulk cake-dyeing practice to use the lowest possible volume of liquor. As a final conclusion, it may be said that package-dyeing machines should be so designed that dyeing can be carried out in a minimum volume, which in the case of cake dyeing appears to correspond to a liquor ratio of about 6 : 1. Furthermore, the maximum possible pump output should be employed short of causing disruption of the cake, so as to obtain the maximum dyebath cycle rate.

Theoretical Model

We now examine the experimental results presented above in the light of the theoretical model previously described by Boulton and Crank [2]. In the absence of evidence to the contrary, they assumed that the rate of uptake of dye by any element of package at a given time is directly proportional to the difference between the amount of dye actually on the yarn at the time and the amount it would take up if left to reach equilibrium with the dye liquor in its immediate vicinity. Calculated skein-dyeing curves based on this and two other assumptions are compared with corresponding measured curves in Fig. 5 and 6. The calculated curves are based on one or other of the following assumptions—

(*a*) The rate of uptake of dye by the yarn is governed entirely by diffusion within the fibres.

(*b*) The rate of uptake is given by the expression—

$$\frac{\partial M}{\partial t} = k_1(RC^m - M) \qquad \text{(i)}$$

where RC^m is the amount of dye which 1 g. of yarn would take up if left to reach equilibrium with liquor containing dye at concentration C, M is the amount of dye actually on the yarn, and k_1 is a rate constant. The constants R and m are characteristic of the absorption isotherm for the dye at the temperature of the dyebath.

(*c*) The rate of uptake is given by the expression—

$$\frac{\partial M}{\partial t} = k_2(RC^m - M)^2 \qquad \text{(ii)}$$

where the various quantities have the same significance as above and k_2 is a second rate constant.

The various constants k_1, k_2, D/r^2 (D = diffusion coefficient of dye in viscose rayon fibres; r = radius of fibres) are chosen so that all curves pass through 50% of final exhaustion at the same time of dyeing. For assumption (*b*) we have take-up of dye by the yarn when RC_m exceeds M, and washing off when RC^m is less than M. For assumption (*c*), however, the rate of washing off is given by $-k_2(RC^m - M)^2$. No mechanism is advanced at the moment to account for either of the expressions (*b*) and (*c*).

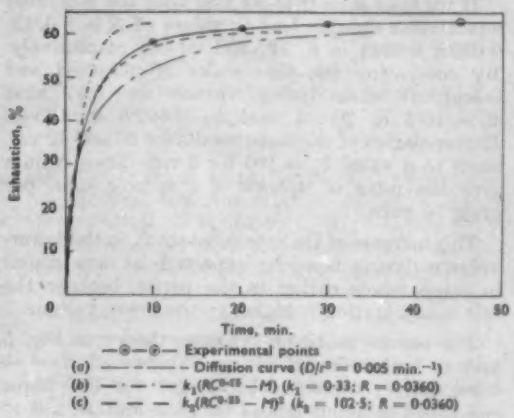
Exhaustion, %
Time, min.
Experimental points
(a) Diffusion curve (D/r^2 = 0·005 min.$^{-1}$)
(b) $k_1(RC^{0.42} - M)$ (k_1 = 0·33; R = 0·0360)
(c) $k_2(RC^{0.33} - M)^2$ (k_2 = 102·5; R = 0·0360)

FIG. 5—Skein-dyeing Curves (12 vol., 63% exhaustion)

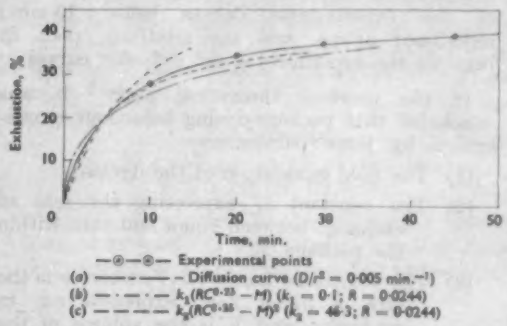
Exhaustion, %
Time, min.
Experimental points
(a) Diffusion curve (D/r^2 = 0·005 min.$^{-1}$)
(b) $k_1(RC^{0.33} - M)$ (k_1 = 0·1; R = 0·0244)
(c) $k_2(RC^{0.35} - M)^2$ (k_2 = 46·3; R = 0·0244)

FIG. 6—Skein-dyeing Curves (20 vol., 42% exhaustion)

The details of the calculation of the curves in Fig. 5 and 6 are discussed in the Appendix. If the dyeing takes place from a bath so large that the concentration C in the bath remains constant as dyeing proceeds (an infinite dyebath), then the expression under assumption (*c*) becomes—

$$\frac{\partial M}{\partial t} = k_2(M_\infty - M)^2 \qquad \text{(iii)}$$

where M_∞ is the amount of dye finally taken up by the yarn in equilibrium. It is interesting to note in passing that the integrated form of this expression is—

$$k_2 t = \frac{1}{M_\infty - M} - \frac{1}{M_\infty} \qquad \text{(iv)}$$

which is the equation of a hyperbola, suggested by Vickerstaff[3] as giving a good fit to many rate-of-dyeing curves.

Inspection of Fig. 5 and 6 reveals that (1) the calculated dyeing curve based on assumption (*c*) is a much better approximation to the measured curve than that based on (*b*) and is good enough for most practical purposes; (2) the simple diffusion curve (based on (*a*)) is not in as good agreement with the experimental curve as that based on assumption (*c*), but it is to be noted that there are no experimental observations below 10 min. on Fig. 5 and 6, and so the experimental curves for times less than 10 min. are ill-defined.

If we take $m' = 0{\cdot}25$ we find that the measured equilibrium uptakes lead to values of $R = 0{\cdot}0513$, $0{\cdot}0360$, $0{\cdot}0244$ in 6, 12, and 20 vol. respectively. By comparing the time-scales of observed and calculated skein-dyeing curves we find that $k_2 = 46{\cdot}3$ in 20 vol. and $k_2 = 102{\cdot}5$ in 12 vol. Extrapolation of the skein results for 20 and 12 vol. leads to a value $k_2 = 106$ for 6 vol. These values give the rates of uptake of dye in g./min. per gram of yarn.

This increase of the rate constant k_2 in the lower-volume dyeing is to be expected, as was stated in other words earlier in the paper, because the salt concentration is higher in the lower volume.

The curves based on diffusion theory in Fig. 5 and 6 both correspond to a value of $D/r^2 = 0{\cdot}005$ min.$^{-1}$, from which, since r for the fibres concerned is about 0·0011 cm., we find $D = 6 \times 10^{-9}$ cm.^{2}/min. This must be regarded as an order of magnitude only, in view of the lack of definition of the experimental curves below 10 min., mentioned above, and the relatively poor fit between the experimental and diffusion curves.

In the previous theoretical paper[2] it was concluded that package-dyeing behaviour is controlled by three parameters—

(1) The final exhaustion of the dyebath

(2) The constant k_1, expressing the rate of exchange between liquor and yarn within the package

(3) The composite factor v/k_1V, where v is the rate of liquor flow, expressed e.g. in cm.^{3}/min., and V is the volume of the dyebath. The greater the value of v/k_1V, the more level is the dyeing at a given time, other things being equal.

With this in mind, we may examine the dyeings listed in Table I, where the amount of salt added was adjusted to keep the final exhaustion approximately the same in the two different volumes. If k_1 had the same value in the two volumes, then clearly v/k_1V in 6 vol. exceeds v/k_1V in 20 vol. for the same rate of flow v. Thus on this assumption the theoretical model previously described[2] suggests that the 6-vol. dyeing will be the more level, as Table I shows to be the case. To produce the same exhaustion in 20 vol. as in 6 vol., however, calls for more salt in the higher volume, and so from what we have said earlier we expect k_1 to have a higher value in the higher volume. This makes v/k_1V in 6 vol. even greater compared with its value in 20 vol. This enhances the rate of levelling on the time-scale of the model, but this is a non-dimensional time-scale given by k_1t, where t is the true time in minutes. Thus the smaller k_1 in the lower volume tends to increase the true time in minutes by virtue of this factor, and so it is not possible to say for certain whether the lower- or the higher-volume dyeing will become level more quickly, without first working out the detailed mathematics of the theoretical model.

It is even more difficult, without detailed calculation, to estimate the effect of the liquor ratio for dyeings in which no salt is added. In this case, as we have seen, k_1 has a greater value in the low volume than in the high volume, and so v/k_1V in 6 vol. may be greater or less than v/k_1V in 20 vol. Furthermore, the final exhaustions are different in the two volumes, and so all three of the basic parameters listed above vary at once. In such a situation only detailed calculations for given conditions can indicate the effect of liquor ratio.

Such calculations have been made using essentially the same method as that outlined in Part I[1]. When assumption (*b*) regarding the rate of exchange of dye within the package was used, it was impossible to fit all the experimental curves of Fig. 3 with a single value of k_1: a value $k_1 = 0{\cdot}1$ was needed for short dyeing times and $k_1 = 0{\cdot}025$ for longer times. This is to be expected from the way in which the skein-dyeing curve, calculated on the same assumption for $k_1 = 0{\cdot}1$, deviates from the measured curve in Fig. 6: the calculated rate is too large as equilibrium is approached. A single value ($k_1 = 0{\cdot}33$) is more successful in 6 vol. than in 20 vol., though here, again, in order to get the correct levelness, a relatively smaller k_1 is required at the end of the package where the liquor enters and where equilibrium is nearly attained very soon after the start of dyeing, and a relatively larger one where conditions are further from equilibrium. Clearly, the simple assumption (*b*), on which the exploratory calculations of the earlier theoretical paper[2] were based, is not adequate for a detailed quantitative comparison in this system.

The agreement between experimental and theoretical curves for skein dyeing was much improved by using assumption (*c*) above, i.e. a rate of uptake of dye given by $k_2(RC^{0{\cdot}25} - M)^2$. In Fig. 7 and 8 this is shown to be true also for package dyeing. The calculated values of percentage exhaustion at successive times agree well with the measured values for both volumes, when the values for k_2 deduced from the skein-dyeing calculations for corresponding volumes are used. Unfortunately, it is not possible to dye a small

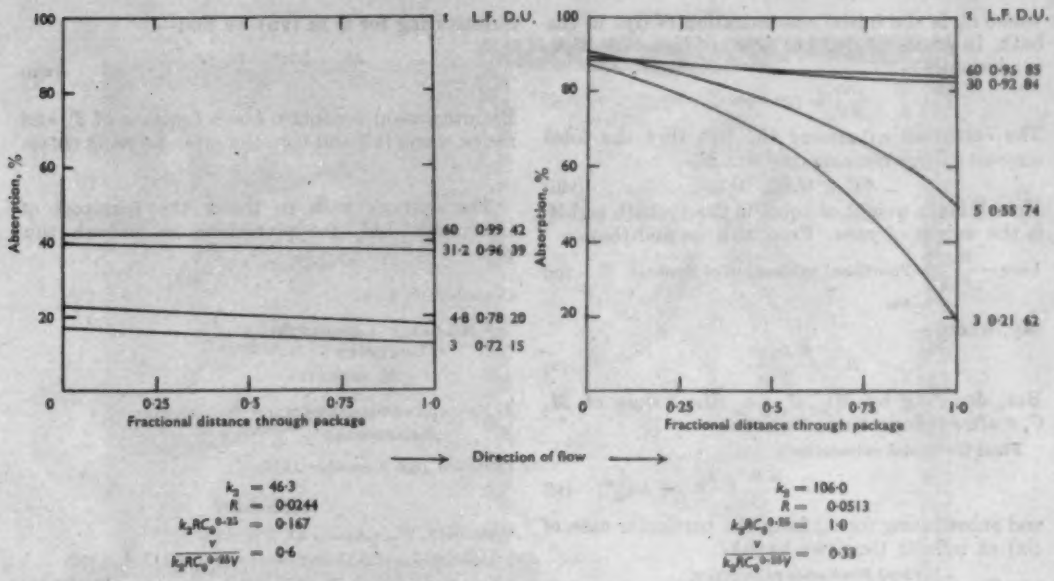

FIG. 7—20-vol. Liquor (46% exhaustion) FIG. 8—6-vol. Liquor (85% exhaustion)

FIG. 7 and 8—Calculated Package-dyeing Curves for $\partial M/\partial t = k_2(RC^{0\cdot25} - M)^2$

skein satisfactorily in 6 vol. in order to make rate-of-dyeing measurements. The lowest convenient volume is 12, and so an extrapolated value for k_2 in 6 vol. is used. In comparing Fig. 7 and 8 with Fig. 3 and 4 it is to be remembered that the experimental results are drawn as straight lines because measurements were made only at the two ends of the package and not at intermediate points. With this in mind, we see that, whilst there is room for improvement in the calculated levelness factors, the general picture is probably as good as can be expected in view of the difficulty of making measurements for a skein in 6 vol. and in view of the simplicity of the theoretical model. The main conclusions already drawn on the basis of the experimental curves are substantiated by the calculations based on the theoretical model, namely that—

(1) In low volumes a higher final exhaustion is achieved more rapidly

(2) The levelness is not as good initially in the low volume, but it rapidly improves and becomes as good as in the higher volume.

For the new rate of exchange, $k_2(RC^{0\cdot25} - M)^2$, the three basic parameters are the same as those listed above for the linear exchange law, except that $k_2RC_0^{0\cdot25}$ replaces k_1 throughout. Here C_0 is the concentration of dye in the bath initially. On looking at the values of the basic parameters given in Fig. 7 and 8, we find that we have a lower exhaustion and a higher value of $v/(k_2RC_0^{0\cdot25}V)$ in 20 vol. than in 6 vol., although v/V is smaller for 20 vol. Both of these factors contribute to the initially more level dyeing in 20 vol., but the subsequent rapid improvement in levelness of the 6-vol. dyeing occurs because $k_2RC_0^{0\cdot25}$ is six times as large in 6 vol. as in 20 vol., and we have noted earlier that the true time of dyeing is inversely proportional to k_1 (or to $k_2RC_0^{0\cdot25}$). We see once again, therefore, that a low exhaustion and a high rate of circulation of the dye liquor produce level dyeings, but that there are two opposing effects associated with the rate of exchange of dye between liquor and yarn: on the one hand, a rapid exchange accelerates the whole process of dyeing, but at the same time it introduces a differential effect tending to unlevelness between one end of the package and the other.

Only detailed calculation can reveal which of these several effects will predominate, but a full investigation of the effects of variations in the three basic parameters is beyond the scope of this work.

Appendix

CALCULATION OF SKEIN-DYEING CURVES FOR A FINITE DYEBATH

In the main paper we have assumed that the rate of exchange of dye between liquor and yarn is governed by an expression of the type—

$$\frac{\partial M}{\partial t} = k(RC^m - M)^n \qquad \text{(v)}$$

Here M is the amount of dye taken up by unit weight of yarn. We have considered only the two cases corresponding to $n = 1$ and $n = 2$. We derive here the formulae on which the calculated curves of Fig. 5 and 6 are based.

Take new variables—

$$c = \frac{C}{C_0} \quad w = \frac{M}{RC_0^m} \quad T = \frac{kt}{(RC_0^m)^{n-1}} \qquad \text{(vi)}$$

where C_0 is the initial concentration of dye in the bath, in grams of dye per gram of liquor, so that (v) becomes—

$$\frac{\partial w}{\partial T} = (c^m - w)^n \qquad \text{(vii)}$$

The condition expressing the fact that the total amount of dye remains constant is—

$$VC + WM = VC_0 \qquad \text{(viii)}$$

Here V is the weight of liquor in the dyebath and W is the weight of yarn. From this we find that—

$$1 - c = \frac{WM}{VC_0} = \text{Fractional exhaustion of dyebath} = Bw \qquad \text{(ix)}$$

say, where—

$$B = \frac{WRC_0^m}{VC_0} \qquad \text{(x)}$$

But, denoting by M_∞, C_∞, c_∞ the values of M, C, c after infinite time, we have—

$$\text{Final fractional exhaustion} = \frac{WRC_0\, c_\infty}{VC_0} = Bc_\infty^m \qquad \text{(xi)}$$

and substituting for c_∞ from the particular case of (ix) at infinite time, we have—

$$B = \frac{\text{Final fractional exhaustion}}{(1 - \text{Final fractional exhaustion})^m} \qquad \text{(xii)}$$

Substituting for w in (vii) we find—

$$-\frac{\partial c}{\partial T} = \frac{(Bc^m - 1 + c)^n}{B^{n-1}} \qquad \text{(xiii)}$$

By integration we obtain c as a function of T, and hence, using (vi) and (ix), the rate-of-dyeing curve.

* * *

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CORRESPONDENCE

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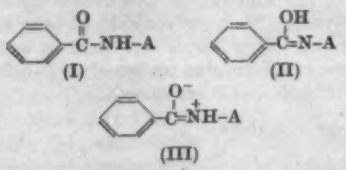
Affinities of Vat Dyes

Peters and Sumner [1] have recently discussed the affinities of vat dyes for cellulose in relation to their chemical structure, and have shown that the affinities can be explained satisfactorily in terms of the abilities of the dyes to form secondary valence bonds with the cellulose substrate. They suggest that there are two main types of bonding forces—(*a*) non-polar or van der Waals forces, and (*b*) dipolar or hydrogen-bond forces. The affinities of over seventy vat dyes can be accounted for in terms of these two mechanisms of dye attachment.

Whilst agreeing with the main conclusions of this excellent study, one may take exception to two minor points of detail. One is the formulation of the benzamido group in the iminol form, and the other is the neglect to take into consideration chelation, or internal hydrogen bond formation, within the dye molecule itself.

The concept of the tautomeric or enol form (II) of the benzamido group, normally represented by the keto form (I), is invoked to explain the much greater affinities of benzamidoanthraquinones compared with, say, acetamidoanthraquinones—



(I) (II) (III)

(A represents the anthraquinone residue). The enol form permits conjugation of the phenyl group with the anthraquinone part of the molecule. However, the concept of an enol form of the amide group has been largely discredited, mainly on the basis of infrared and X-ray structural studies [2]. Still, it is widely accepted that the carbon–nitrogen link does have a partial double-bond character in many amides, and the amide group is probably best regarded as a resonance hybrid of the keto (I) and dipolar (III) forms. Interaction between the benzamido group and the rest of the molecule would still be possible on this view.

With reference to the second point, Peters and Sumner point out that substitution in the phenyl ring of the benzamido group alters the affinity of the dye. Chloro, methyl, and methoxyl groups *meta* or *para* to the amide group invariably cause an increase in affinity, but the same substituents in the *ortho* position always result in a lowered affinity. They suggest that, as a result of steric action by the *ortho* substituents, the phenyl group is forced out of the plane of the rest of the molecule, and thereby the amount of hydrogen bonding, and also the area available for van der Waals attractive forces, are reduced.

These effects may well operate, but an alternative or additional factor is probably important, viz. the internal chelation of the amide group to the *ortho* substituent. The idea that chelation may be important arises from some recent work that I have been doing on the solvent power of various phenols for polyamides [3]. Phenols are, in general, good solvents for polyamides, interacting with the amide group by hydrogen bonding. However,

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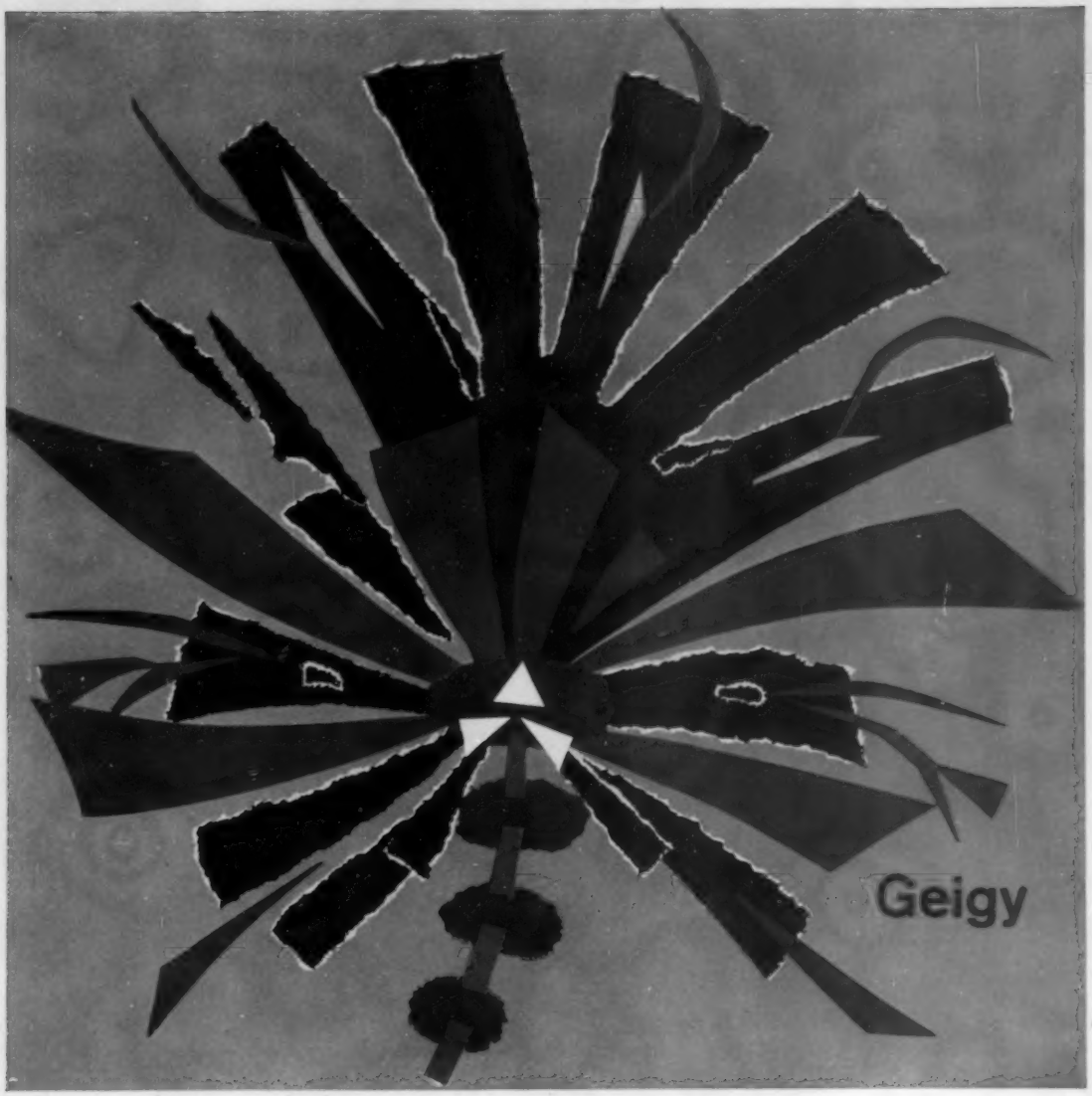

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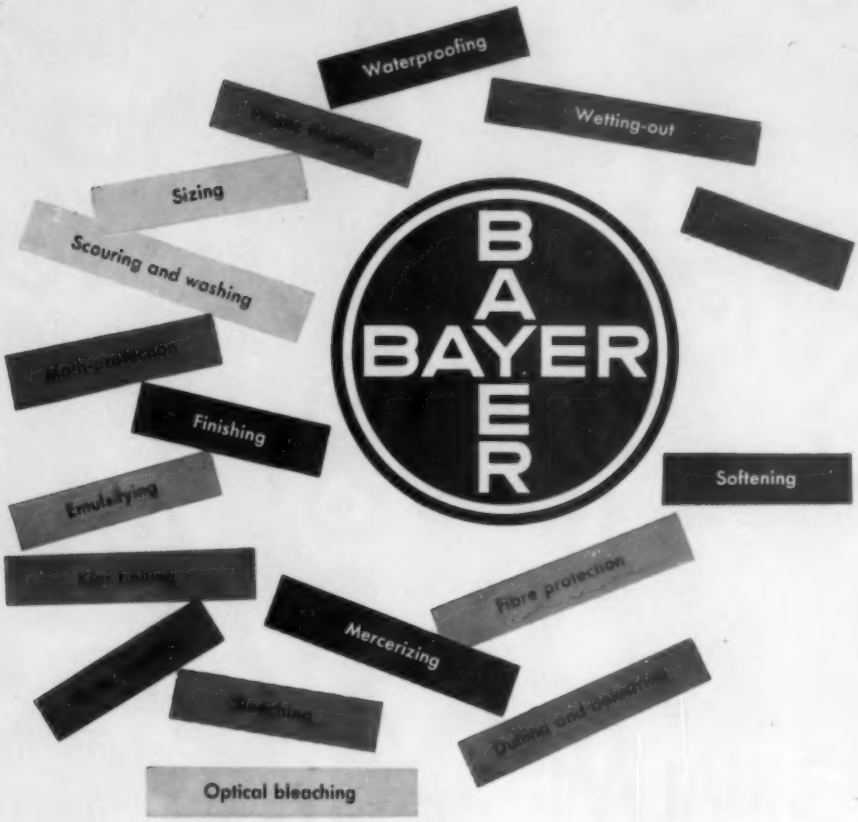

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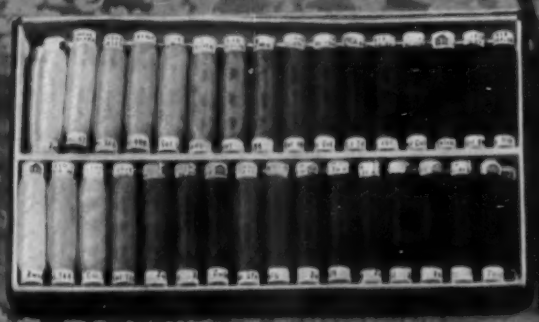

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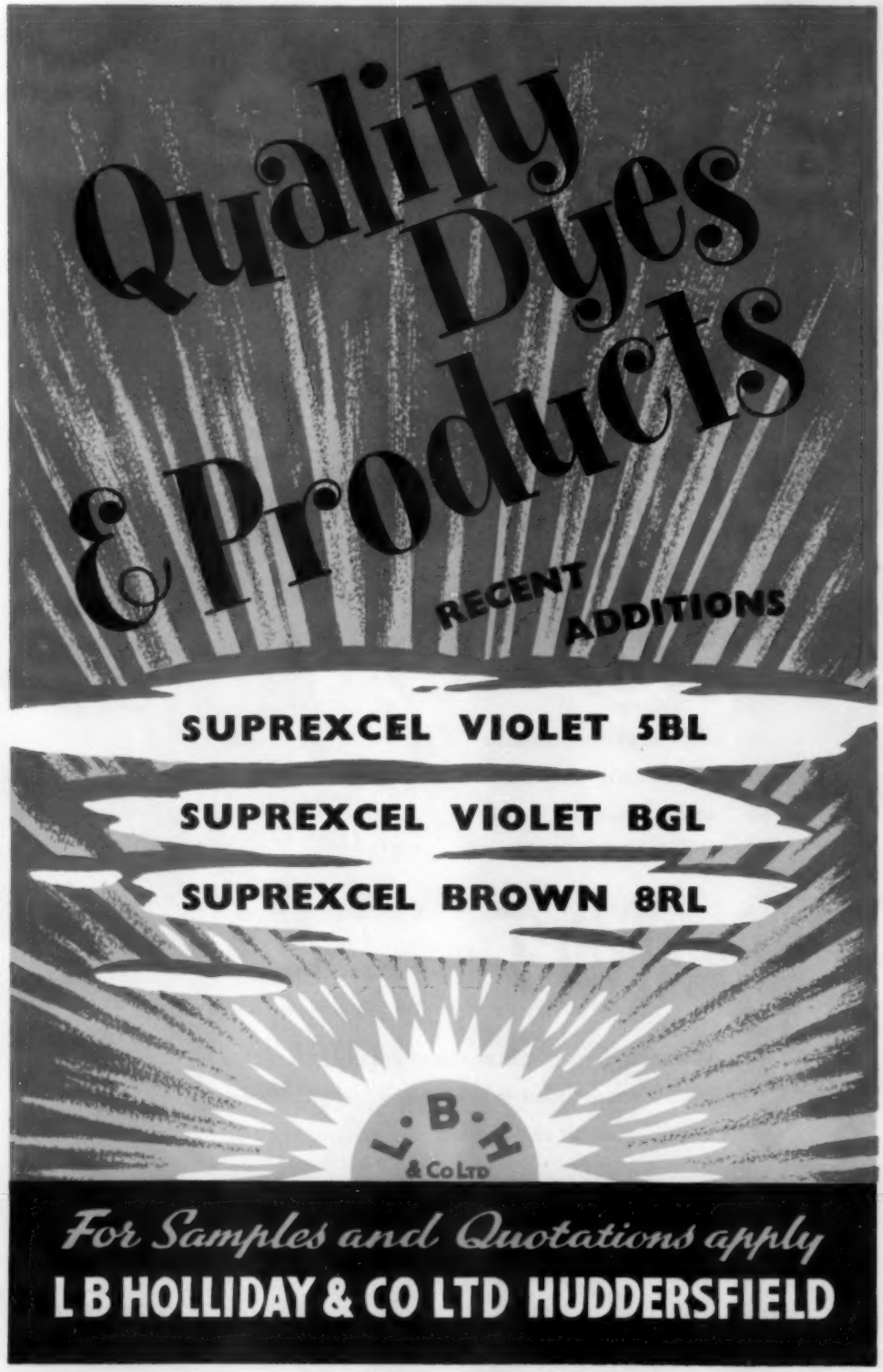
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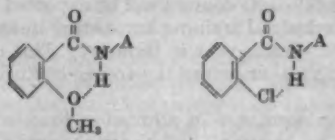
phenols containing *ortho* methoxyl groups are non-solvents, and those with *ortho* chloro groups are poor solvents. These are the very groups which, when *ortho* to the amide group, are most effective in lowering the affinities for cellulose of the benzamidoanthraquinones, and it seems probable, or at least possible, that a common cause is operating.

Now, it is well known that in phenols *ortho* groups such as chloro and methoxyl chelate to the hydroxyl group, which is thus less likely to interact with another molecule. The poor solvent power for polyamides shown by such phenols is thus easily explained.

It is highly probable that benzamido compounds will show similar effects, e.g.—



This chelation will reduce the affinity of the dye, either by reducing the hydrogen bonding between the amide group and the cellulose, or by making the dipolar form, and hence conjugation with the rest of the molecule, less likely.

From the data in the paper of Peters and Sumner, it follows that the effectiveness of *ortho* substituents in lowering the affinity decreases in the order methoxyl > chloro > bromo > methyl. On the basis of purely steric (bulk) effects, the order would have been expected to be Br > Cl > CH_3 > O·CH_3. Although the methoxy group is large, the free rotation about the ether linkage reduces its effective hindering capacity. The greater effectiveness of the methoxyl and chloro groups in lowering the affinity is in harmony with the idea that chelation is important, as the methyl group is incapable of chelation, and the bromo group should show weaker chelation than the chloro group. The behaviour of *o*-fluorobenzamidoanthraquinone would be an interesting test case. On purely steric grounds it should have little or no effect, as its van der Waals radius is too small. On the other hand, as fluorine is more electronegative than chlorine, and the bond lengths are more favourable, chelation would be expected to be much stronger with fluorine than with chlorine, and the affinity should be greatly reduced.

Attention is being drawn to this point because it is believed that the possible influence of chelation should always be borne in mind whenever hydrogen bonding is likely, and that this concept may be useful in future studies of the synthesis of dyes.

LESLIE VALENTINE

DEPARTMENT OF TEXTILE INDUSTRIES
THE UNIVERSITY
LEEDS 2

23rd March 1956

[1] Peters and Sumner, J.S.D.C., **71**, 130 (1955).
[2] Bellamy, *The Infrared Spectra of Complex Molecules* (London: Methuen & Co. Ltd. 1954), pp. 180 and 188.
[3] Valentine, *J. Polymer Sci.*, in the press.

Classification of Dyes by their Dyeing Characteristics

In reading W. Beal's paper on this subject, I have noticed what I think is an important error in the section on direct cotton dyes [1]. In his generous reference to the early work of Boulton and Reading, Mr. Beal states that the time of half-dyeing has provided recorded results showing a four-hundred-fold variation. In fact, they showed a four-*thousand*-fold variation, the most rapidly diffusing dye (Chlorazol Fast Orange GS) having a time of 0·07 min., and the most slowly dyeing (Chlorazol Fast Pink BKS) 280 min.

A number of workers have referred to this very great variation since the appearance of the original work, and I would be glad if you could draw this error to the attention of the author of this latest paper, and to your readers.

JOHN BOULTON

RESEARCH LABORATORY
COURTAULDS LTD.
CAMPBELL STREET
DROYLSDEN
MANCHESTER

26th April 1956

[1] Beal, W., J.S.D.C., **72**, 154 (April 1956).

Effect of Organic Solvents on Wool Dyeing

We were interested and somewhat surprised to read that Dr. L. Peters and Dr. C. B. Stevens [1] consider the basic reaction of applying metal-complex dyes from aqueous ethyl alcohol solutions given in the Swiss patent of 1948 to be completely different from the application of metal-complex dyes from aqueous *n*-butyl alcohol solutions described in the Diploma Thesis of 1954–1955.

Example 4 in *GP* 832,593, in particular, is quite clearly a dyeing from an organic solvent, and in view of the alcohol : water ratio used, it would appear to us to be more accurate to say that water is added to reduce the solubility of the dye in the organic solvent rather than that the alcohol is added to increase the solubility of the dye in the water.

The novelty of the more recent work seems to lie not in any new reaction or mechanism of dyeing but in being able to obtain a high concentration of solvent in the place where it is most needed, i.e. at the wool surface; whereas formerly in the Ciba work the solvent had been dispersed uneconomically throughout the whole dyeing system.

Peters and Stevens have used solvents containing polar groups in order to achieve this surface concentration effect. Our own solution of the same problem has been to use non-polar compounds, which are more readily removed from the fibre.

The history of the Leeds University work from 1954 is certainly of interest when it is considered alongside the following [2]—

> It is not claimed that the most effective solvent or method of use of a particular solvent has been discovered nor have the economic factors been fully considered . . . .
>
> The obvious problems such as removal of solvent after dyeing, the risk introduced by the use of solvents which may be toxic and give rise to dermatitis, and the effect on effluent disposal have not been considered either. That is the business of the dye maker and the dye user . . . .

The partition of work between industry and university appears to be similar to that of dye between butyl alcohol and water in Fig. 1 [2] and justifies the title of the article: "A New Approach to Dyeing".

To the many problems Peters and Stevens have passed to technologists to solve, may we add this: if an equilibrium is established between wool and dye at 50°C., a new one involving change of shade will be established if higher temperatures are used in subsequent processing.

For Sandoz Products Ltd.
G. H. Lister

152 Canal Road
Bradford 2
Yorkshire

4th May 1956

[1] Peters, L., and Stevens, C. B., J.S.D.C., **72**, 241 (May 1956).
[2] Idem, *Dyer*, **115**, 329 (2nd March 1956).

Notes

Meetings of Council and Committees

May

Council— 2nd

Colour Index Editorial Panel— 7th and 24th

Publications— 21st

Examinations Board— 28th

Colour Index

Mr. H. Blackshaw and Mr. C. O. Clark, joint co-ordinators of the *Colour Index* Editorial Panel, met Mr. W. D. Appel, of the A.A.T.C.C., on Thursday, 10th May 1956, in Manchester, when various matters relating to the publication of the Second Edition of the *Colour Index* were discussed.

Deaths

We regret to report the loss by death of Messrs. H. V. Dyson, V. G. Nair, S. Shaw (of Halifax), and C. P. Tattersfield.

Chairman of the Publications Committee

At the meeting of the Publications Committee held on 15th May 1956, Mr. C. L. Bird was elected Chairman in succession to Mr. J. Barritt. Mr. Bird has been closely concerned with the *Journal* for many years, as Assistant Editor, Editor, and also Chairman of the Publications Committee (1949–1953).

Richard Charles Oakley
High Sheriff of Bedfordshire

A member of the London Section and a former Mayor and Alderman of Luton, Mr. R. C. Oakley, has been chosen by the Queen as High Sheriff of Bedfordshire.

Diploma in Technology

The recently created National Council for Technological Awards has announced that it will grant a Diploma in Technology, the courses for which will be equivalent in standard to honours degree courses of a British university. The award will be denoted by *Dip. Tech.* (*Eng.*) for engineering, and by *Dip. Tech.* for other technologies. There will be two honours classes, first and second class, and a pass award.

The courses which will be recognised may be either full-time or sandwich. The minimum time required for academic students in a technical college must not be less than three years full-time, or four years if the course is sandwich. Students attending full-time courses will be expected to have suitable industrial training amounting in aggregate to not less than a year in industry. This must be undertaken either before the course or during it or both.

The minimum age of admission to courses will normally be 18 years, and the standard of admission to courses will generally be that of the General Certificate of Education with two appropriate subjects at advanced level and three subjects at ordinary level.

Suitable courses are expected to include a thorough education in the fundamentals of science and technology and their application to development and design. They must include also liberal studies and some instruction in the principles of industrial organisation.

Teaching is to be conducted in an environment where advanced studies are the main preoccupation of the staff. The college will be expected to provide good library facilities and good social amenities, and it is desirable that it shall have residential facilities, though this last condition will not be insisted upon at first.

Some Implications of Colour
London, 20th September 1956

A one-day symposium with the above title is to be held by the Oil and Colour Chemists Association at University College, Gower Street, W.C.1. Three papers will be given—

Colour Gamuts of Pigments — E. Atherton and D. Tough

Assessment of Light Fastness — J. G. Gillan

Personal Experience with the Inter-society Colour Council Aptitude Test — M. Hess

Full details will be obtainable from the O.C.C.A., Memorial Hall, Faringdon Street, London E.C.4.

British Patents Abridgments

Group volumes of specifications accepted during the 1939–1945 war period are now on sale, so that a complete range of volumes up to and including the *BP* 640,001–660,000 series is now available. Abridgments in sheet form are available for specifications published up to August 1955.

Among subject groups, Group IV(*c*) covers dyes and dyeing, and Group VIII includes textile finishing.

The volumes in bound and sheet form can be obtained from the Sale Branch, Patent Office, 25 Southampton Buildings, London W.C.2, at £1 1s. 0d. to £1 10s. 0d. per group.

Canadian Pigments

Imperial Paper & Color Corpn. of Glen Falls N.Y. has acquired a plant in St. Johns, Quebec, where a Canadian subsidiary, now being formed, will manufacture a range of chemical pigments.

C.O.C.

Indian Dyemaking Industry

A survey of the Indian dyemaking industry is now being made by a team of six Indian and six Russian experts.

Licences have been granted for the manufacture in Bombay of phthalocyanine pigments (7,000 lb. per month), phthalocyanine derivatives (9,000 lb.), and solubilised vat dyes (12,000 lb.).

C.O.C.

Dye Manufacture in Argentina

On 16th March 1956 there was inaugurated in the Llavollol province of Buenos Aires the works of Anilsud Fabrica Argentina de Anilinas S.A., which has been erected by the Compania Química S.A. in co-operation with the German Farbenfabriken Bayer A.G., which supplied the plant and technical advice. It is going to concentrate on dyes for textiles, and is expected to satisfy in full Argentina's annual requirements of 110 million lb.

C.O.C.

Carbon Black in Australia

Work has begun on a £2,000,000 project for the manufacture of carbon black at Geelong, Victoria, Australia. It is designed to produce 16,000 tons per annum, and will be the only carbon black plant in the southern hemisphere.

Perlon (6-Nylon) in Hungary and the U.S.A.

A plant is to be constructed at the Magyar Viscosa works at Nyergesujfalu in Hungary for the manufacture of 6-nylon from caprolactam, which is to be made from phenol at the Hungária Chemical Works.

In the U.S.A. the Allied Chemical & Dye Corpn. is marketing 6-nylon under the names of *Caprolan* for the textile trade and *Plaskon* for the plastics industry. It is made from phenol via *cyclo*hexanone, its oxime, and caprolactam.

Second International Congress of Surface Activity

This is to be held in London during 8–12th April 1957. The membership fee is to be £4. Enquiries should be addressed to the Honorary Secretary of the Congress, 14 Belgrave Square, London S.W.1.

Trade with the U.S.S.R.

Among goods which the U.S.S.R. would like to purchase from the United Kingdom are dyeing and finishing machinery for the textile industry and plant for the production of Terylene.

Musée de l'Impression sur Étoffes, Mulhouse

The buildings were destroyed during the 1939–1945 war, but the collections were saved. After protracted negotiations it has now been agreed that the collections will be on permanent exhibition to the public at 3 Rue des Bonnes-Gens. The executive committee of the Museum will consist of representatives of the Société Industrielle, the municipality, and the chamber of commerce of Mulhouse.

New Books and Publications

Chemical Pilot Plant Practice

By Donald G. Jordan. Pp. viii + 152. New York and London: Interscience Publishers. 1955. Price, $3.50.

Pilot plants are in many ways the most interesting type of plants, whether from the engineer's, the chemist's, or the operator's point of view. They present many diverse and individual problems, operational, design, and economic; and they are rarely amenable to the same treatment as full-scale plant. It is therefore a pleasure to read a book devoted entirely to this subject, the more so since few textbooks give much space to it.

The present book does not set out to be more than a review of pilot-plant principles and practice, and, as the author says in his preface, is somewhat biased in the direction of the synthetic organic chemical industry. The work is intended primarily for chemical engineers, and being American in origin refers essentially to transatlantic practice. The approach is that of the practical man rather than the theorist, which adds to the interest and the value of the subject-matter. This is arranged in six chapters, the first dealing with definitions and general considerations, while the remainder deal with the important specific topics of scale-up, materials handling, chemical reactors, separation processes, and cost estimation and report writing respectively. It may come as a surprise to some readers that the shortest chapter in the book is that on chemical reactors.

It is difficult to single out any part of this book for special mention, but there is little doubt that many engineers will find the chapter on scale-up very good value indeed. An excellent brief outline is given of the problems; and the methods available for tackling them, based on the principles of similarity and the theory of models, are indicated with examples. A short but adequate bibliography is provided, although it seems a pity that the author does not refer at all to the long series of articles which have appeared over the years in *Industrial and Engineering Chemistry* describing individual pilot plants; although obviously specific, these can often be very helpful.

The author writes clearly and concisely, and his meaning is never in doubt: actually, the conciseness of style is perhaps excessive, being achieved at the expense of readability in places. Possibly, however, the fault is due rather to the continual use of numbered paragraphs and sentences. A few well chosen illustrations supplement the text, and provide further evidence of the author's care in preparing his book.

There seems little doubt that this volume will become a well thumbed reference book for many engineers—and for many plant chemists. It will also be of great value to students, who will be particularly grateful to the author for his brief but clear explanations. Some knowledge of pilot plant is obviously mandatory for Branch 9 (Production of Dyes, Lakes, and Pigments) of the A.S.D.C. Examination Paper D (Industrial Procedures and Plant); but much of this book is applicable to the other branches of this paper also, in spite of the word "Chemical" in the title.

I. R. McDougall

ASTM Standards on Soaps and Other Detergents

(with related information)

Prepared by ASTM Committee D-12 on Soaps and Other Detergents. Pp. viii + 163. Philadelphia: American Society for Testing Materials. 1955. Price, paper, $2.50.

The new edition of this book is a collection of standards from the much larger Part 7 of the *ASTM Book of Standards*. The field covered is the same as the last edition (1952), with corrections, revisions, and a small number of additions proposed since that time.

Seventeen standard specifications are presented for twenty-three soap products, together with six standard specifications and three tentative specifications for alkalis. Analytical and other test procedures are given for these, and for soap products containing synthetic detergents, sulphated and sulphonated oils, and metal-cleaning agents, including methods for foaming properties, pH value, surface and interfacial tension, buffering action, and corrosive action of the relevant types of cleaning agent.

New contents include tentative standards for the analysis of sodium triphosphate. Specifications for soda ash, sodium metasilicate, and trisodium phosphate have been revised. The rinsing, buffering, and some corrosion tests previously published as "proposed" are now tentative standards. Comments, offered by your reviewer on the 1952 edition, have received attention; the large errors in the specification for sodium bicarbonate have been corrected and "alkaline detergents" have been redefined as *inorganic alkaline detergents*.

For a book of standards, proofreading and quality of printing should be faultless, and should support standards which themselves command the highest respect. Unfortunately, a number of imperfections appear, some of them inherited from previous issues.

The instruction to dilute concentrated sulphuric acid *with* water is twice given, without any word of caution, in the details for the Liebermann–Storch test for rosin (pp. 56 and 78). In the interests of safety, these directions should be rewritten to conform with the more correct method of dilution used elsewhere.

Weighing instructions tend to be ambiguous and inconsistent. "Weigh 5 $\pm$ 0·01 g. . . . " (p. 50) appears to call for between 4·99 g. and 5·01 g., although from the text it is clear that it is not essential to be within this range to obtain accurate results. On the next page, in "Digest 2 to 10 $\pm$ 0·01 g. . . . " the $\pm$ convention seems to mean that any quantity between 2 g. and 10 g. weighed to the nearest 10 mg. meets the requirement. The estimation of rosin by the McNicoll method is presented three times, in which the same detail is given twice as "Weigh about 2 $\pm$ 0·001 g. . . . " and once as "Weigh 2 $\pm$ 0·001 g. . . . ". In all, some eleven different forms of instruction for accurate weighings are given in which the necessary tolerances are not always specified.

In the rosin estimation calculation (pp. 56 and 132) the definition of the term "A" is irrelevant: on p. 43, P_2O_5 appears as PO_2O_5. In the copy submitted for review, the significant figure for Free Acid in Type B Olive Oil Chip Soap fails to appear, and on p. 77 the decimal point in the equation $R_1 = R - 1{\cdot}0$ does not reproduce.

These defects detract from the value of the book, which, nevertheless, represents a useful assembly of testing procedures with a wider scope than standards yet proposed in Great Britain.

D. F. B. Kevan

Leather Finishes

Their Formulation, Manufacture, and Application

By J. S. Mudd. 2nd edition 1955. Pp. viii + 141. Croydon: A. Harvey. Price, 15s. 0d.

Perhaps the most striking advances in connection with leather manufacture which have taken place in the past twenty years have been in the methods of finishing leather by the application of dyes, pigments, cellulose and other films, and many other materials. Manufacturers of leather not only have taken advantage of the use of new methods such as synthetic resins, but also have adopted new techniques in applying older and more established materials. Until fairly recent times, leather

manufacturers had to rely largely on the use of materials and methods which had been designed specifically for use on other materials. For instance, very few dyes had been synthesised for particular use on leather. Leather is such a complex, woven fibrous material that it is quite fortuitous whether a material or method which is ideal for another fibre, say cotton or wool, is equally suitable for leather. Moreover, there are so many variations in leather, depending not only on the kind of animal or even the part of the animal from which the skin was taken, but also on the method of tanning employed, that no single method or material is universally applicable in the finishing of leather. Recently, leather has come more into its own in the choice of suitable materials and methods for converting tanned leather into the finished article for so many diverse uses. Much research work and experimentation have taken place in the preparation and application of finishing materials.

No-one is better qualified to describe modern methods and the most recent developments than the present author, having himself specialised for many years in this field. The present work has an introductory chapter specially designed for the "non-leather man", giving an outline of the terms and phrases used in describing the many processes in leather manufacture. Then follow sections describing the sources and characteristics of many of the raw materials used in the processes of leather finishing, with special emphasis on pigments. Individual chapters deal with the raw materials used for the two main types of pigment finishes for leather, namely water finishes and cellulose finishes. The vexed question as to how much pigment should be applied to the surface of leather, that is, from the slight amount needed merely to obtain, say, more levelness of colour or fastness to light, to the heavy application which obliterates most of the natural appearance and feel of leather, is quite fairly and honestly dealt with.

As colour plays a predominant part in the appraisal of a finished leather, the chapter on colour measurement by modern scientific methods is very welcome. This field is largely unfamiliar to leather men.

The method of preparing finishes suitable for leather and of applying them are treated in such a way as to be of great value not only to the student of the technology of leather manufacture but also to those actually engaged in putting the finishing touches to the material which "there is nothing like".

J. R. BLOCKEY

A Review of Calibration of Fading Lamps used in Canada

A Study by the Canadian Association of Textile Colourists and Chemists Committee on Test Methods. Pp. iv + 14. Montreal: Textile Technical Federation of Canada *Technical Bulletin* No. 2. September 1955. Price, $1.00.

In 1950 the C.A.T.C.C. sent a piece of direct-dyed spun rayon staple material to 25 laboratories for determination of (amongst other properties) its light fastness. The results showed very wide variations: most of the laboratories employed the normal American procedure of expressing light fastness in terms of the number of hours of exposure in a fading lamp necessary to produce an easily perceptible fade, and the results varied from 4 hr. to 40 hr. The Committee on Test Methods therefore decided to investigate this problem in detail, and its results have now been published.

The method employed was to send a piece of the lamp calibration paper of the United States National Bureau of Standards to each lamp operator with the request that it be exposed for 20 hr.; a detailed questionnaire was also submitted which asked for relevant details concerning lamp-operating conditions.

The N.B.S. supplies standard faded strips of the calibration paper which have been exposed for varying periods to the light of the master lamp in Washington, and it is therefore a simple matter to determine the relative speed of any lamp. The Committee found that Canadian lamps varied considerably, the slowest providing only 12 standard fading hours in 20 clock hours, whilst the fastest produced 28 S.F.H. in the same time.

The Committee concluded that, if any degree of uniformity in determining colour fastness to light is to be achieved, an adjustment in the fading rate of about 60% of the lamps now in use in Canada would be required.

K. MCLAREN

Laundry Chemistry

London: British Launderers' Research Association. 1955. Pp. vii+152. Price, 12s. 6d.

This book is intended primarily for students of laundry technology and for technicians in the laundry industry. It may come as a surprise to some that this industry provides work for 140,000 people, and although technical training has not developed as rapidly as in some other industries, considerable efforts are being made to remedy this, since it is felt that, like the textile industry, the laundry industry of the future will require more and more scientifically trained workers. One difficulty has been the lack of a textbook concerned primarily with the scientific bases of washing materials and methods, since *The Chemistry of Laundry Materials* by D. N. Jackman (London: Longmans, Green & Co. 1931) has long been out of print.

The present volume is intended to provide an up-to-date successor to Jackman and follows the same general plan. The language and the style are simple, and no prior knowledge of chemistry is assumed, although some acquaintance with science is desirable. The book opens with a long chapter on general chemistry, which includes the laws of chemical combination, valency, acids, bases, and salts, ionisation, pH, hydrolysis, buffering power, and a brief account of the simpler aliphatic organic compounds. A chapter on volumetric analysis follows, in which detailed attention is given to the analysis of water supplies, boiler waters, wash liquors, rinse waters, and stock alkali solutions.

The "laundry chemistry" section proper comprises ten chapters dealing with water, alkalis, detergency, soaps, synthetic detergents, bleaches, stain removal, acids, blues, and finishing agents. Recommended testing methods are included in an appendix.

Unlike Jackman, this book contains no detailed account of textile materials, since it is considered that textbooks already exist which cover this subject adequately at all levels. However, as this book is intended for students, this omission may be unwise. The present volume is the work of several members of the staff of the British Launderers' Research Association, in particular G. R. Perdue and F. R. Hill, and they are to be congratulated on the result of their efforts, which should meet a real need. The style is easy, the printing and the layout are good, and the price is very reasonable indeed.

C. B. STEVENS

British Standard Methods for Determining the Colour Fastness of Textiles

B.S. 2661–2686 : 1956

Pp. 68. London: British Standards Institution. Price, 12s. 6d.

This compilation consists essentially of test methods first published in the *Third Report of the Fastness Tests Co-ordinating Committee* (J.S.D.C., **71**, 283–342 (June 1955)) and subsequently as *Standard Methods for the Assessment of the Colour Fastness of Textiles*, now appearing in the format of a British Standard.

The twenty-six individual standards relate to—principles of colour fastness testing, grey scale for assessing change in colour, grey scale for assessing staining, and colour fastness to the following twenty-three agencies—acid spotting, alkali spotting, bleaching with hypochlorite, bleaching with peroxide, carbonising with aluminium chloride, carbonising with sulphuric acid, acid chlorination, mercerising, chromium salts in the dyebath, iron and copper in the dyebath, organic solvents, spotting, hot pressing, rubbing, soda boiling, stoving, washing by hand, water, sea water, water spotting, washing (Test No. 2 (wool)), washing (Test No. 4 (natural and regenerated cellulose)), and acid milling. All except the last three tests are proposals of the international subcommittee (ISO/TC 38/SC 1).

The method for determining colour fastness to daylight (also an international proposal) is given as an appendix. It is the same test as *B.S.* 1006, although different in presentation.

C. J. W. H.

British Scientific Instruments 1956

Directory and Handbook of S.I.M.A.

London: Scientific Instrument Manufacturers' Association of Great Britain Ltd. Pp. cxliv + 268. No price.

This directory should prove of great assistance to those who are contemplating the not inconsiderable outlay entailed in increasing the "instrumentation" of their dyehouses or other works. It consists of a directory of member firms of S.I.M.A., a classified index of their products with glossaries in French, German, and Spanish, and illustrated "announcements" of members. The directory is not to be regarded as a complete catalogue, but enquiries concerning apparatus not mentioned will be referred by S.I.M.A. to a suitable source of supply.

C. J. W. H.

Methoden der Organischen Chemie (Houben–Weyl)

Band III

Physikalische Methoden

Teil 1

Edited by Eugen Müller. 4th edition 1955. Pp. xxix + 954. Stuttgart: Georg Thieme Verlag. Price, DM 162.

This third volume of the well known Houben–Weyl work on methods of organic chemistry deals with those physical research methods which may be broadly classified as mechanical, thermal, microscopic, mass-spectrographic, and isotopic. Under these headings are grouped sections, each contributed by an expert or experts, dealing with related methods.

A first section on thermodynamic methods gives an introductory account of classical thermodynamics, methods of calculating chemical affinity, entropy changes, and equilibrium constants, and a brief account of intermolecular forces, much of which is to be found in standard texts. It is followed by a useful section on kinetic methods, in which the determination of reaction velocity and the interpretation of data are discussed and consideration is given to complex systems such as those involving chain and simultaneous reactions. Sections on the determination of density, solubility, and vapour pressure follow. There are separate sections on determination of molecular weights of substances of low and high molecular weight. The latter includes a good account of the light-scattering method. Under viscosity methods it is perhaps a little surprising to find that, although some Staudinger constants are given, values of K and a in the Mark–Houwink relation are not. The section includes a brief discussion of polymolecularity. There is a useful section on surface tension and surface activity, and an account of calorimetric methods which includes consideration of the determination of specific and latent heats, heats of mixing, solution, and dilution, and heats of reaction. Vapour space effects in heats of mixing and the use of thermistors for determining temperature changes do not seem to be mentioned. A very good section is concerned with the use of atomic models and another with the estimation of errors in physical measurements. There is a large section on microscopic methods, which includes an account of the geometrical properties of crystals, the preparation of substances for microscopic study, and the determination of double refraction. This is followed by a general account of mass-spectrographic methods and a section on the use of radioactive isotopes in which general methods are discussed and the uses of specific isotopes briefly considered. A final section deals with the analytical estimation and uses of non-radioactive isotopes

and includes estimation by density, optical, spectrographic, and other methods.

References up to the end of 1954, and in some cases to work published in 1955, are given, and there are well chosen bibliographies at the end of each section. There are many useful tables, and diagrams are clear. Binding and printing are good. While a few methods cited may be, perhaps, of historical interest only and treatment is, in a few cases, rather brief, the book bears comparison with others of a similar type. Both physical and organic chemists will find many data and much useful information in this work.

W. R. MOORE

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes.
Any publication abstracted may be referred to by members of the Society on application to
Dr. C. B. Stevens, Dyeing Department, Leeds University

The Clayton Dyestuffs Co. Ltd.

NAVY BLUES ON WOOL PIECE GOODS— This card contains dyeings in three depths of ten dyes from the Kiton and Neolan ranges suitable for producing navies on woollen and worsted cloths, including blazer cloths, dress cloths, and outerwear. Fastness figures have been determined according to the recommendations given in the *Second Report of the Society's Fastness Tests Committee*, and details of the test methods are included.

The Geigy Co. Ltd.

DYESTUFFS FOR POLYAMIDE FIBRES— This card contains dyeings in two depths on yarn of the following dyes suitable for polyamide fibres—14 Novalon dyes, 9 level-dyeing acid dyes, 13 acid dyes applied from weakly acid dyebaths, 27 neutral-dyeing acid dyes (25 from the Polar range), 4 Irganol S dyes, 18 Irgalan-type metal-complex dyes, and 13 Eriochrome dyes. The Novalon dyes are disperse dyes, applied in presence of soap or Eriopon AC as dispersing agent and a small amount of trisodium phosphate. They are particularly level dyeing even on barry material and are recommended for dyeing all types of goods where only moderate fastness to light and wet treatments is demanded.

ERIO ANTHRACENE BRILLIANT BLUE F3GL—This level-dyeing acid dye is recommended for use alone and in mixtures on wool yarn and cloth. Dyeings have good fastness to light, perspiration, and salt-water spotting, and they maintain their hue in artificial light. Fastness figures on wool include— Light 5–6, perspiration (S.D.C. test) 3, washing (S.D.C. test No. 1) 3.

ERIO FAST FLOXINE KL; ERIO FAST FLOXINE NHE— These are level-dyeing acid dyes for dyeing wool yarn and cloth from a sulphuric acid–Glauber's salt dyebath to give bright reds. Dyeings have good fastness to light, carbonising, and decatising. Their most interesting characteristic, however, is their stability to prolonged boiling, which makes them very valuable for use alone and in mixtures for dyeing yarn in enclosed machines; the standard of colour reproducibility attainable is considerably higher than hitherto associated with dyes of the Floxine type. Their excellent levelling properties combined with the very good fastness of dyeings to carbonising make them particularly suitable for dyeing wool cloth. Fastness figures on wool include — *Erio Fast Floxine NHE:* Light 5, carbonising 5; *Erio Fast Floxine KL:* Light 5–6, carbonising 5.

SETACYL DYESTUFFS ON ACETATE RAYON— This card contains dyeings in two depths on knitted acetate rayon fabric of 31 disperse dyes covering the colour range from yellow to navy blue, and also three direct dyeing and two diazotised-and-developed blacks. A more comprehensive range of fastness figures than given formerly is included, and also conversion tables for British and metric measures, percentage dye into weight, and comparative temperatures.

Imperial Chemical Industries Ltd.

PIGMENTS FOR PAINT: ORGANIC AND INORGANIC— This card describes 27 Monolite and 2 Monastral pigments, 15 toners, 4 madder lakes, 4 maroon lakes, 3 barium and zinc chromes, 11 lead chromes, 7 Prussian Blues and Brunswick Greens, and a special green, Chromastral Green MS, a coprecipitated mixture of a lead chrome and a phthalocyanine blue giving a non-floating green brighter in hue than the Brunswick Greens. The lead chromes include the more recently developed types, which do not darken on exposure to light. Fastness data include light in linseed oil (Madder Scale, 1–10) and in alkyd and nitrocellulose compositions (*B.S.* 1006, 1–8), solvents, heat and acid, alkali, lime, water, and linseed oil. Information is given for matching colours given in *B.S.* 381C : 1948, *Colours for Ready Mixed Paints:* three alternatives are given in each case— (*a*) the best all-round recommendation, non-bleeding in common paint and lacquer solvents; (*b*) tending to bleed in solvents but superior to (*a*) in light fastness or other properties; (*c*) an alternative non-bleeding combination.

VERILAN DYES FOR LOOSE WOOL AND SLUBBING— This card contains dyeings in four depths on wool slubbing of 12 individual dyes and 24 mixtures. The Verilan dyes are a new range of metallisable dyes which are self-developing. They are particularly recommended for pale to medium colours on loose wool and slubbing where the fastness demands are comparable with those normally demanding the use of chrome dyes. The dyeings obtained are somewhat brighter than when the latter are used, and since they are self-developing and have a slow rate of strike, a simple and relatively quick-dyeing method may be used. Thus dyeing is started at 50°C., the temperature raised to 95–100°C. as rapidly as possible, and dyeing continued at the boil for 45 min. If conditions permit, dyeing may be started at the boil. For pale to medium dyeings addition of 2–5% ammonium sulphate is sufficient, but for deep dyeings a small amount of acetic acid may be required to exhaust the dyebath.

WATER-SOLUBLE THIONOL DYESTUFFS M BRANDS— This card contains dyeings in two depths on cotton drill of 12 solubilised sulphur dyes constituting the M range. Their freedom from insoluble matter and their level-dyeing properties enable the dyer to exercise a degree of control not possible with the conventional types, and they are preferred for dyeing yarn in packages and thick, tightly woven, or nappy cotton cloths. They are completely soluble in water without any further treatment, but sodium sulphide must be added to convert them into a form having affinity for the fibre.

Sandoz Products Ltd.

ACID ALIZARINE BLACK R— This chrome dye is recommended for dyeing wool yarn and knitted and woven cloth by the afterchrome process to give dyeings of good fastness to light, washing, and decatising. It is very level dyeing, and cellulosic effects are reserved white. It is sensitive to both iron and copper. Fastness figures include— Light 6, carbonising 5, alkaline milling 3–4.

SOLAR COLOURS STABLE TO SIZING ON PAPER— This card describes five direct dyes from the Solar range recommended for colouring paper. These dyes are unaffected by sizing, and the hue and the depth of the colourings obtained are the same whether the dye is added to the pulp before or after sizing. The light fastness obtainable with Solar dyes is higher than with the fastest acid dyes; they are also less sensitive to the degree of sizing. Colourings in four depths of the five dyes are included to show the effect of sizing before and after adding the dye, and also ten mixture colourings to show that these dyes are insensitive to sizing also when used together.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Thread-storing, Thread-advancing Reel
American Viscose Corpn. *USP* 2,707,668

Measuring the Surface Temperature of Rollers, etc. or of Cloth, Paper, or Yarn passing through a Machine
Mason-Neilan Regulator Co. *USP* 2,712,236

Continuously Drying Rayon Cakes
American Viscose Corpn. *USP* 2,709,856

Handling Material in Rope Form with Maintenance of Constant Tension
Proctor & Schwartz *BP* 746,311
The material is fed on to a tapered roll, along which it is free to move. An elongated restraining member parallel to the roll engages the material. The tapered roll is driven at constant speed, the rate at which the material is advanced being thus determined by the radius of the roller at the point of engagement, and the latter being automatically determined by the tension in the material between the roll and the restraining member. C.O.C.

Circulating Dye Liquor through One or More Vats or the Like
Burlington Engineering Co. *USP* 2,707,382
Apparatus for circulating dye liquor from vat to vat and also within each individual vat. This enables a large number of pieces to be dyed to an exact match in separate vessels. C.O.C.

Dyeing Fabric on Beams
Burlington Engineering Co. *USP* 2,707,383
The cloth is wound on to a foraminous beam and lowered into a dye vat, where the ends of the beam fix into saddles forming part of the vat sides. One end of the beam is engaged by an adjustable sealing gasket, which thrusts the other end of the beam into sealing engagement with a fixed gasket. The dye liquor circulates through one end of the beam, and through the wound cloth into the vat. The beam is stationary throughout dyeing. C.O.C.

Hydroextracting by Radial Compression
Mezzera *BP* 746,923
The material is piled on a perforated central support and prevented from expanding at either end. Pressure set up in the annular chamber surrounding the material compresses it, and expels the water in it through the holes in the support. C.O.C.

Web Tension Control
Research Corpn. *USP* 2,710,153–4
Means for ensuring that the tension in a web after it has been unwound from, or before it is wound on to, a roll is independent of that in it when it is being unwound or wound. C.O.C.

Drying Cloth, Paper, etc.
Svenska Flaktfabriken AB. *BP* 746,244
A machine in which the hot air or other drying medium is supplied so that the different stages of drying take place under different physical conditions in order to obtain the utmost efficiency. C.O.C.

Raising Machine
Parks & Woolson Machine Co. *USP* 2,707,815
Method for controlling the cloth-feeding and -positioning mechanism. C.O.C.

Treatment of Bias Fabrics
Gates Rubber Co. *USP* 2,709,475
Method by which the angle of the warp to the weft can be accurately controlled while keeping the weft threads straight and parallel to one another. It is particularly suitable for use in conjunction with the application of rubber to produce calendered fabrics. C.O.C.

Felt
L. S. Adams Engineering *USP* 2,708,303
Machine for the rapid felting in open width of batts, especially those containing large amounts of non-feltable fibres. C.O.C.

Screen Printing Machine
J. H. Stokes *USP* 2,709,404
Means for ensuring even pressure of the squeegee and for enabling very large repeats to be printed. C.O.C.

Renovating Furs and Pile Fabrics
R. S. Schaab and J. E. Tiso *USP* 2,712,230
A wide carrier conveys the fur or fabric to a hot ironing roller. The carrier is so arranged that in the case of garments one strip after another is treated to remove dust, dirt, and loose hair or fibre from near the rapidly rotating roller without danger to either the material being treated or the operator. C.O.C.

Modern Methods of Electrostatic Flocking of Textiles, Paper, etc. (X p. 309)

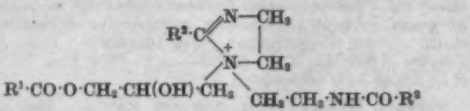
III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Colloidally Dispersed Oxides for the Textile Industry
K. Dithmar
Textil Praxis, **10**, 1134–1137 (Nov. 1955)
Colloidal silica is widely used in various types of finishing agents, softeners, and anti-static agents, and also for anti-slip finishes on polyamide fibres. Colloidal alumina has a positive charge and therefore can be used with cationic and non-ionic auxiliary products in contrast to colloidal silica. It is used for matt and other finishes and is of special interest for non-slip finishes, where it can be combined with a cationic softener such as Sapamine KW (Ciba). Colloidal titanium dioxide is used as a white pigment in fibre manufacture. B.K.

PATENTS

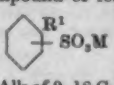
Textile Lubricant
Textile Oils *BP* 746,742
Method of making emulsions, containing blown or unblown sperm oil, suitable as carding and spinning lubricants, which are readily removed in scouring. C.O.C.

Cationic Surface-active Agents
American Cyanamid Co. *USP* 2,708,666
An epihalogenohydrin is used to link a hydrocarbon radical of a cyclic carboxylic acid with the N atom of a polyalkylenepolyamine, to form a halide salt of an amino-ester containing the radical $R^1{\cdot}CO{\cdot}O{\cdot}CH_2{\cdot}CH(OH){\cdot}CH_2-$ attached to the polyamine N atom, e.g. a quaternary ammonium cpd. of formula—

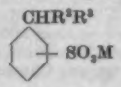


(R^1 and R^2 = hydrocarbon radicals of a tall-oil fatty acid). C.O.C.

Detergents
Esso Research & Engineering Co. *USP* 2,712,530
A mixture of a compound of formula—



(R^1 = branched-chain Alk of 9–18 C; M = alkali metal) with 10–100% of its weight of a compound of formula—



(R^2 = straight-chain Alk of 1–5 C; R^3 = straight-chain Alk of 8–14 C; $R^2 + R^3$ = 9–15 C) is a much more effective detergent at low concn. than the additive effect of both compounds alone. C.O.C.

Sequestering Agents
Dow Chemical Co. *USP* 2,712,544

Compounds of formula—

$$R\cdot[NH\cdot Alkylene]_n\cdot NH_2$$

(R = Alk containing the $-\overset{O}{\overset{\diagup\diagdown}{C}}-C-$ group; $n > 1$; at least one amino H is displaced by $CH_2\cdot COOM$ or $CH_2\cdot CH_2\cdot COOM$ (M = alkali metal); Alkylene = an alkyl group which adds 2–3 C to the chain between the N atoms), e.g.—

$$\overset{O}{\overset{\diagup\diagdown}{CH_2}}\text{-}CH\text{-}CH_2\cdot CH_2\cdot N(CH_2\cdot COONa)\cdot CH_2\cdot CH_2\cdot N(CH_2\cdot COONa)_2$$

are active metal-ion-chelating agents in aq. soln. at pH > 7 and in alcohol, ether, and liquid hydrocarbons, in which solvents they have pronounced dehydrating properties. C.O.C.

Salts of Epoxy Dicarboxylic Amino Acids
Dow Chemical Co. *USP* 2,712,545

Compounds of formula $R\cdot NH_4$ (R = Alk, preferably of 10–20 C, containing the $-\overset{O}{\overset{\diagup\diagdown}{C}}-C-$ group; at least one H is replaced by an acid group, preferably $CH_2\cdot COOM$ or $CH_2\cdot CH_2\cdot COOM$; M = alkali metal, ammonium, or an amine), e.g.—

$$\overset{O}{\overset{\diagup\diagdown}{CH_2}}\text{—}CH_2\cdot CH_2\cdot N(CH_2\cdot COONa)_2$$

are sequestering agents soluble in water, alcohol, ether, and liquid hydrocarbons. C.O.C.

Polymeric Titanium Acylates—Waxes, Surface-active Agents, Lubricants, etc.
DuP *USP* 2,708,203

The products obtained by heating a mixture of a polymeric titanium acylate ("carboxylate") and a carboxylic acid of b.p. above that of the carboxylic acid coproduct corresponding to the acyl radicals of the original titanium acylate, and distilling off the coproduct, are viscous liquids to waxy solids, useful as waxes, surface-active agents, etc. C.O.C.

Hydroxymethylacetone Resin Precondensate—Resin Finishes
Calico Printers' Assocn. *USP* 2,711,971

Strict limitation of the alkali-metal hydroxide to the highly critical range of 0·025–0·1 mol. of hydroxide per mol. of acetone results in exhaustion of the alkali and stopping of the reaction at the precondensate to yield a methylacetone precondensate free from vinylacetones and polymers thereof. There are 4 mol. of H·CHO per mol. of acetone. Used in resin finishing, the product gives a full shrink-resisting finish without imparting stiffness to the fabric. C.O.C.

Composition for imparting Permanent Set to Hair
Horizons *USP* 2,707,697

A solution of a zirconium compound, preferably in a solvent containing organic hydroxyl and carboxyl groups, is used; e.g. zirconium sulphate, oxychloride, or nitrate (5 parts) and citric acid (2) in warm water. C.O.C.

Allylstarch Coating Composition for Application to Keratinous Material—Insectproofing
Merck & Co. *USP* 2,707,689

Allylstarch dissolved in toluene is formed into a concentrated aqueous emulsion using sodium lauryl sulphate as the emulsifying agent. This emulsion is used for coating keratinous material, particularly wool cloth, which has been previously treated with a triethanolamine silicofluoride insecticide, this rendering the insecticide fast to washing and dry cleaning. C.O.C.

Stable Aqueous Silanol or Vinylsilanol Dispersions
Libbey-Owens-Ford Glass Co. *BP* 746,037

An aqueous solution of an alkali-metal or alkaline-earth metal siliconate is brought to pH 3–7 by treatment with the hydrogen form of a cation exchanger. The siliconate has 0·05–3·0 organic groups attached to each Si atom, these groups being uni- or bi-valent saturated or uni-valent olefinically unsaturated hydrocarbons of 1–5 C or uni- or bi-valent aromatic groups of 6–7 C. They are used as water-repellent agents, e.g. for glass fibres. *BP* 746,038

An aqueous solution of an alkali-metal or alkaline-earth metal vinylsiliconate is brought to pH 3–8 quickly enough to prevent precipitation of the resulting silanol. The siliconate has 0·05–3·0 organic groups attached to each Si atom; of this on the average 0·05–1·0 are vinyl groups, the others being saturated hydrocarbon groups of 1–5 C or aromatic hydrogen groups of 1–6 C. They are used as water-repellents, particularly for glass fibres. C.O.C.

Drying Emulsions for Treating Textiles, Leather, etc.
Ciba *BP* 746,394

Emulsions containing a metallic drier and as binder an aminotriazine–formaldehyde condensate (aminotriazine containing > 1 NH_2, condensate in which at least 2 CH_2OH groups per mol. of aminotriazine are etherified by allyl or methallyl groups) are used for producing finishes, prints, etc. on textiles required to be fast to rubbing and washing. C.O.C.

Germicidal and Antistatic Composition for Treating Fabrics
J. L. Campbell, A. H. Osmond, and G. C. Hutchinson *USP* 2,709,665

A bacteriostatic and anti-static agent for use on fabrics, especially those of cotton and linen, which also renders them less liable to dust or shed lint, comprises, by volume, U.S.P. white mineral oil (50–80), emulsifying agent (1–25), wetting agent (1–5), cationic germicide (2–6), and 1–16% aq. NH_4F,HF (1–20). C.O.C.

Trifluoroacetic Acid as Solvent for Polyethylene Terephthalate
Eastman Kodak Co. *USP* 2,710,848

Terylene and similar polyesters yield clear solutions in trifluoroacetic acid, as much as 33 g. of the polymer being soluble in 100 g. of the acid. C.O.C.

Dimethyl Methanephosphonate as Solvent for Polyacrylonitrile
Chemstrand Corpn. *USP* 2,710,847

Dimethyl methanephosphonate is an excellent solvent for polymers containing ≮ 70 % of acrylonitrile. C.O.C.

Tanning Agents of Good Fastness to Light
Progil *BP* 745,626

A primary formaldehyde–*p*-cresolsulphonic acid condensate is treated with a primary formaldehyde–phenol or –4:4′-dihydroxydiphenyl sulphone condensate in presence of acid, using 0·7–1·5 mol. of *p*-cresolsulphonic acid to 1 mol. of phenol, or 2 mol. of the sulphonic acid to 1 mol. of the sulphone. C.O.C.

Metal-complex Azo Dyes as Printing Assistants in the Application of Thioindigoid Vat Dyes (IX p. 306)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Dyes and Dyeing in Ancient Mesopotamia
M. Lerey
J. Chem. Educn., **32**, 625–629 (1955); *Chem. Abs.*, **50**, 3763 (10 March 1956)
71 references. C.O.C.

Emilio Noelting (1851–1922)
J. Lichtenberger
Teintex, **21**, 126–135 (Feb. 1956)
A short biography with records of Noelting's contributions to dye chemistry. S.R.C.

Richard Herz
A. Lüttringhaus
Chem. Ber., **89**, I–X (March 1956)

A. Lüttringhaus
H. Neresheimer
Chem. Ber., **89**, XI–XVII (March 1956)

Directive Effects in Electrophilic Aromatic Substitution
K. LeRoi Nelson
J. Org. Chem., **21**, 145–155 (Feb. 1956)

Aromatic Nucleophilic Substitution
IV—Relative Nucleophilic Powers of Common Reagents
C. W. L. Bevan and J. Hirst
J.C.S., 254–260 (Feb. 1956)

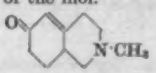
V—Influence of the Solvent
C. W. L. Bevan and G. C. Bye
Ibid., 469–472

Comparison of the Stability of Some Intramolecular Hydrogen Bonds
H. Hoyer
Chem. Ber., **89**, 146–150 (Jan. 1956)

From infrared spectra of five appropriate cpd. it is concluded that hydrogen bonds from hydroxy groups which give rise to six-membered rings are more stable than that to chlorine, which yields a five-membered one. Groups *ortho* to a hydroxy substituent give increasingly stable hydrogen bonds in the order I, Br, Cl, (F), NO_2, CO, –N:N–. H.E.N.

Decahydro*iso*quinolines and Related Compounds. I— Some 6-Oxygenated Derivatives and an Example of Abnormal Ultraviolet Absorption
A. Marchant and A. R. Pinder
J.C.S., 327–331 (Feb. 1956)

The synthesis of 1:2:3:4:6:7:8:9-octahydro-2-methyl-6-oxo*iso*quinoline (I) is described; it gives the maximal ultraviolet absorption at an abnormally short wavelength, viz. 227·5 mμ. ($\varepsilon = 10{,}000$), and it is suggested that the N atom, although not directly attached to the chromophore, raises the energy associated with the electronic disturbances therein, so that maximum ultraviolet absorption occurs at a shorter wavelength. A dipolar structure such as II may make an important contribution to the stable state of the mol.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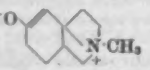

(II) H.H.H.

By-products of the Alkaline Hydrolysis of Chlorobenzene and the Mechanism of their Formation
A. Lüttringhaus and D. Ambros
Chem. Ber., **89**, 463–474 (Feb. 1956)

The residues from the distillation of the phenols from the high-pressure process are shown to consist of 4-hydroxydiphenyl (93%), 3-hydroxydiphenyl (<0·3%), a high-boiling fraction (4%), and solids, mainly hydrocarbons (1%). The "neutral" residue of the distillation of diphenyl ether consists of 2-hydroxydiphenyl (30%), 2:6-diphenylphenol (9%), 2-phenoxydiphenyl (45%), 4-phenoxydiphenyl (~8%), bound sodium (2·9%), and tarry–glassy residues (4%). The formation of these products is explained by analogy with the interaction of diphenyl ether with phenyl-sodium or -potassium. H.E.N.

Novel Method of Cyanoethylation. IV— Substitution Effects
R. J. Bates, J. Cymerman-Craig, M. Moyle, and R. J. Young
J.C.S., 388–395 (Feb. 1956)

Preparation of *N*-2-cyanoethylarylamines, by amine exchange from 2-diethylaminoethyl cyanide (I) and the arylamine, is shown to depend on the basic strength of the arylamine and to be hindered by *ortho* substitution and also by bulky *N*-substituents in the arylamine. Instead of preformed I, a mixture of vinyl cyanide and diethylamine may be used. The effect of methyl substitution in the cyanoethyl group itself points to a substitution mechanism. The nature of the amine displaced also affects the yield, diethylamine giving optimum results. The methiodides of dialkyl-2-cyanoethylamines undergo exchange with cyanide ion to give 1:2-dicyanoethane. H.H.H.

1-Phenylnaphthalenes. III— Effect of Substituents and their Position on the Absorption Spectra of 1-Phenylnaphthalene-2:3-dicarboxylic Anhydrides
F. G. Baddar and Z. Sawires
J.C.S., 395–400 (Feb. 1956)

Structures proposed for some alkoxy-1-phenylnaphthalene-2:3-dicarboxylic anhydrides are shown to be supported by their absorption spectra, now described. Shifts in the absorption bands occur with change in position of the alkoxyl group. The importance of directional effects in connection with the absorption of light is shown. H.H.H.

Reactions of *p*-Chlorobenzenesulphonic Acid and its Derivatives. The σ_p^* Constant of the *NN*-Dimethylsulphamoyl Group
E. L. Eliel and K. W. Nelson
J. Org. Chem., **20**, 1657–1665 (Dec. 1955)

The acid is the principal by-product of the manufacture of DDT from chlorobenzene and chloral in presence of sulphuric acid. Its dimethylamide is converted into *p*-dimethylsulphamoylphenol by means of 10% aqueous NaOH at 200°C. in 4 hr. and into the dimethylamide of sulphanilic acid in 63% yield by ammonolysis at 250°C. H.E.N.

Sandmeyer Reactions of Monoacylarylenediamines
E. D. Bergmann and M. Bentov
J. Org. Chem., **20**, 1654–1656 (Dec. 1955)

From the appropriate acylated phenylenediamine the following cpd. were prepared: *N*-phthaloyl-*o*-aminobenzonitrile (83% yield); 2-amino-5-methoxybenzonitrile (63%), *p*-iodo- (51%), *p*-bromo- (47%), *p*-cyano- (64%), *m*-bromo- (41%), and *m*-cyano-acetanilide (72%). The acyl group was phthaloyl in the second example. H.E.N.

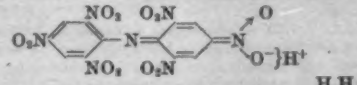
Aromatic and Heterocyclic *o*-Dialdehydes. I— Ready Synthesis of Naphthalene-2:3-dialdehyde
W. Ried and H. Bodem
Chem. Ber., **89**, 708–712 (March 1956)

Interaction of 2:3-dimethylnaphthalene and *N*-bromosuccinimide in presence of benzoyl peroxide leads to 2:3-bisdibromomethylnaphthalene, which is easily hydrolysed, best with aqueous–alcoholic potassium oxalate. Derivatives of the resultant dialdehyde are prepared. Naphthalene-1:2-dialdehyde cannot be obtained analogously. H.E.N.

Colour Digest. Colour Developers and Couplers— Review of Patents
K. M. Hornsby
Brit. J. Phot., **102**, 606–608 (1955)

Spectrophotometric Study of Hexanitrodiphenylamine (Dipicrylamine). I— Molecular and Ionic Forms in Dioxan
S. Kertes and J. M. E. Goldschmidt
J.C.S., 401–404 (Feb. 1956)

Hexanitrodiphenylamine, in its yellow (I) and red (II) forms, when investigated potentiometrically and spectrophotometrically in dioxan soln., is found to be a strong acid, and its red form to be an anion of an *aci*-nitro acid. The K salt, which is insol. in water, is found to be a simple salt in dioxan. The intense colour is ascribed to the continuous system of conjugated double bonds, and so the possibility of ionic resonance in the anion, since both *p*-nitro groups and all four *o*-nitro groups are equivalent insofar as each can become the *aci*-nitro group.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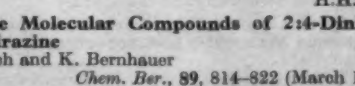
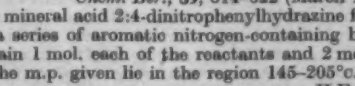



(II)

H.H.H.

Crystalline Molecular Compounds of 2:4-Dinitrophenylhydrazine
W. Friedrich and K. Bernhauer
Chem. Ber., **89**, 814–822 (March 1956)

In dilute mineral acid 2:4-dinitrophenylhydrazine forms cpd. with a series of aromatic nitrogen-containing bases. These contain 1 mol. each of the reactants and 2 mol. of acid. All the m.p. given lie in the region 145–205°C. H.E.N.

Steric Relations between Ionisation of Aralkyl Chlorides and Dissociation of Anilinium Ions— II and III
G. Baddeley, J. Chadwick, and H. T. Taylor
J.C.S., 448–450 and 451–456 (Feb. 1956)

II— Rates of ethanolysis of several tertiary aralkyl chlorides RR′CAr·Cl, when compared with the dissociation constants of the corresponding anilinium ions RR′NArH⁺, indicate that ionisation of the chlorides and dissociation of the anilinium ions effect changes of intramolecular conformation which increase intramolecular steric interaction.

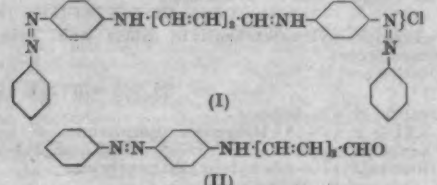
III— Rates of solvolysis of the chlorides $C_6H_5\cdot CCl\cdot[CH_2]_{n-5}$ and of their *o*-methyl deriv. in ethanol, where $n = 5$–7, and the basic strengths of the corresponding amines $C_6H_5\cdot N\cdot[CH_2]_{n-1}$ and of their *o*-methyl deriv. in 50% aq. ethanol, show that the reactivity of the chlorides decreases in the order $n = 5 > 7 > 6$, and, since this is the order of increasing basic strength of the amines, it would appear to be the order of increasing steric hindrance of ionisation of the chlorides and of dissociation of the anilinium ions. Also the reactivity of the chlorides $o\text{-}C_6H_4\cdot CHCl\cdot[CH_2]_{n-6}$ decreases in the order $n = 5 > 6 > 7$, which is the order of increasing basic strength of the corresponding amines $o\text{-}C_6H_4\cdot NH\cdot[CH_2]_{n-5}$ and of their *N*-methyl deriv., thereby indicating this to be the order of increasing steric hindrance of ionisation of the chlorides. H.H.H.

Formation and Reductive Scission of Bis Compounds from Enols
W. Bradley and L. J. Watkinson
J.C.S., 319–327 (Feb. 1956)

The following phenols when heated with azobenzene may undergo substitution or self-union with loss of hydrogen (either one or two atoms per mol.): anthrone gives 9:9′-dihydro-9:9′-dianthrone, 2-methyl-1-naphthol affords 3:3′-dimethyldi-1-naphthyl-4:4′-quinone, and α-naphthol yields 2-anilino-1:4-naphthaquinone 4-anil; 4-methyl-1-naphthol gives the same anil with 1:2-di-(3-anilino-1:4-dihydro-4-oxo-1-naphthylidene)ethane. Analogous cpd. result from 3:3′-dimethyl- and 3:3′-dibromo-azobenzene. Hitherto no self-union of this type has been reversed, but it has now been found that 1:1:2:2-tetrabenzoylethane can be reduced to dibenzoylmethane by Mg in hot or aq. pyridine. Amongst analogous cpd. only 1:2-dibenzoyl-1:2-di-*p*-toluoylethane behaves similarly; others afford furan deriv. or do not react. Mg subiodide also gives furans, but reductive fission is not observed either with this reagent or with Raney Ni and H_2. H.H.H.

Structure and Colour of *p*-Phenylazo-substituted Anils of Glutaconaldehyde
N. E. Grigor'eva and R. M. Zueva
J. Gen. Chem. U.S.S.R., **26**, 107–111 (Jan. 1956)

1-(2:4-Dinitrophenyl)pyridinium chloride reacts with *p*-aminoazobenzene with formation of the pentamethin dye I. Thermal decomposition of I results in elimination of one mol. of *p*-aminoazobenzene and recyclisation of the residue to 1-*p*-phenylazophenylpyridinium chloride, the pyridine ring of which is re-opened by alkali with formation of II. The absorption spectra of I and II are determined in various solvents in presence of acids and bases, and the variations observed are interpreted on the basis of salt formation and tautomeric change.



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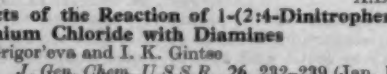
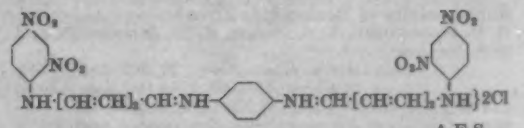


(II)

A.E.S.

Products of the Reaction of 1-(2:4-Dinitrophenyl)-pyridinium Chloride with Diamines
N. E. Grigor'eva and I. K. Gintso
J. Gen. Chem. U.S.S.R., **26**, 232–239 (Jan. 1956)

Nine further "pyridine" dyes (see J.S.D.C., **70**, 366 (1954) and previous abstract) are prepared by the action of 1-(2:4-dinitrophenyl)pyridinium chloride on *o*-, *m*-, and *p*-phenylenediamines and their deriv., reaction occurring between 1 and 2, 1 and 1, or 2 and 1 of the respective reactant molecules, depending on conditions. Their absorption max. are in the range 480–600 mμ. Various new pyridinium salts are prepared from the products by the reaction described previously (*loc. cit.*). The dyes are of the symmetrical 1 : 2 type already described (*loc. cit.*), of an analogous but unsymmetrical 1 : 1 type in which one terminal radical is 2:4-dinitrophenyl, and of the symmetrical 2 : 1 type, e.g.—



A.E.S.

Microsublimation of Azoic Bases on a Hot-stage Microscope
W. Kunze
Textil Praxis, **11**, 160–162 (Feb. 1956)

The use of a hot-stage microscope for the observation of the cryst. form of azoic bases is described. The physical appearance, both before and after sublimation, sublimation range, and m.p. of a large number of azoic bases are tabulated. B.K.

Relationship between the Structure of an Azotol [Azoic Coupling Component] and its Substantivity
B. M. Bogoslovskii
J. Appl. Chem. U.S.S.R., **29**, 152–154 (Jan. 1956)

The 4-diphenylylamide, 4′-nitro-4-diphenylylamide, and 4-phenylazoanilide of 3-hydroxy-2-naphthoic acid are synthesised and dyed on cotton. The extents of exhaustion (%) under the conditions used are 18·6, 21·6, and 38·4 respectively (cf. the anilide, 10·5), and therefore increase with increase in the length of the conjugated system of bonds. A.E.S.

Separation of Alcohol Esters of 4′-Nitroazobenzene-4-carboxylic Acid
El Sayed Amin and E. Hecker
Chem. Ber., **89**, 695–701 (March 1956)

Aromatic Diazo Compounds. XIX— Coupling of *m*-Phenylenediamine with Diazobenzenesulphonates
F. Mužík and Z. J. Allan
Chem. Listy, **49**, 212–218 (1955): *Collection Czechoslov. Chem. Commun.*, **20**, 623–630 (1955): *Chem. Abs.*, **50**, 1641 (10 Feb. 1956)

Coupling of $m\text{-}C_6H_4(NH_2)_2$ (I) with 2 mol. of *o* (II), *m* (III), and *p* (IV)-$N_2\cdot C_6H_4\cdot SO_3^-$ was studied to determine the proportions of monoazo dye (V), vicinal (VI) and symmetrical (VII) bisazo dyes, and trisazo dyes (VIII) produced with variation in pH. The following results were obtained (coupling component, medium of coupling, % of V, VI, VII, and VIII)— II, $NaHCO_3$, 26, 39, 9, 26; III, $NaHCO_3$, 45, 7, 5, 43; IV, acid, 33, 30, 3–4, 33; IV, $NaHCO_3$, 27, 34, 5, 34; IV, Na_2CO_3, 20, 16, 48, 16; $C_6H_4\cdot N_2\}Cl$, dil. acetic acid, 6, 22, 39, 33; $C_6H_4\cdot N_2\}Cl$, Na_2CO_3, 7, 21, 54, 18. C.O.C.

Coal-tar Colours. XV— FD&C Red No. 1, D&C Red No. 5, and Some Related Colours
L. S. Harrow and J. O. Milham
J. Assocn. Off. Agric. Chem., **38**, 172–182 (1955): *Chem. Abs.*, **50**, 2175 (10 Feb. 1956)

Preparation of pure samples of FD&C Red No. 1 (di-Na 1-(3:4:6-trimethylphenylazo)-2-naphthol-3:6-disulphonate) and FD&C Red No. 5 (di-Na 1-(2:4-dimethylphenylazo)-2-naphthol-3:6-disulphonate) is described. The following di-Na 1-(alkylated phenylazo)-2-naphthol-3:6-disulphonates may be present as impurities in commercial samples of these two dyes— 2:5-dimethyl, 2:4:6-trimethyl, 2:6-dimethyl, 2:3-dimethyl, 2-ethyl, and 4-ethyl. Spectrophotometric curves for the visible range (General Electric recording spectrophotometer) are given for all the above in 0·1N-HCl and 0·1N-NaOH (in some cases in 0·04N-$CH_3\cdot COONH_4$). The infrared curves (KBr pellets; Perkin–Elmer Model 21 recording spectrophotometer) are also given. In neutral solution these dyes are orange–red to bluish red and conform to Beer's law. C.O.C.

Basic Azo Derivatives of Thiazole— III
G. Modena
Gazz. chim. ital., **85**, 922–925 (1955): *Chem. Abs.*, **50**, 2175 (10 Feb. 1956)

When the dyes 2-(arylazo)thiazole (I), 2-(arylazo)benzothiazole (II), 2-(arylazo)naphtho[2,1]thiazole (III), and 2-(arylazo)naphtho[1,2]thiazole (Ar = dimethylaminophenyl, diethylaminophenyl, 1-aminonaphthyl, or 2-aminonaphthyl) are applied to cellulose acetate and nylon, the intensity of the dyeings diminishes from I to IV. Dyes II and III on nylon show good fastness to light, washing, and perspiration. C.O.C.

Substantivity of Benzanilide Dyes
B. M. Krasovitskii, V. A. Blinov, R. M. Matskevich, and O. S. Slavina
Ukrain. Khim. Zhur., **20**, 392–395 (1954): *Chem. Abs.*, **50**, 3763 (10 March 1956)

The dyes R·N:N·C_6H_4·CO·NH·C_6H_3·N:N·R′ (R′ = 1-amino-8-hydroxy-3:6-naphthalenedisulphonic acid or R acid) have greater affinity for cotton than three similar dyes (derived from *p*-aminobenz-2:4-diaminoanilide, and phthalic and naphthalic phenylamides respectively) none of which had an H atom in the amide group. It is suggested that the H atom in the amide group is very mobile, and splits off as a proton during dyeing in alkaline solution. Rearrangement of the electrons then occurs with formation of an anion having a long chain of conjugated double bonds. C.O.C.

Ultraviolet Absorption Spectra of Substituted Pyrazoles
D. S. Noyce, E. Ryder, and B. H. Walker
J. Org. Chem., **20**, 1681–1686 (Dec. 1955)

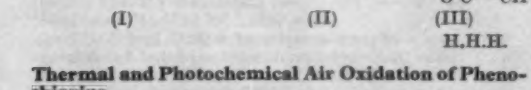
Reaction of Benzoquinonemonoimine with Lignin. II—Isolation and Identification of the Colouring Matters formed
J. Gierer
Chem. Ber., **89**, 257–262 (Feb. 1956)

Prototropy of Crystal Violet
W. Broser
Z. Naturforsch., **100**, 350–355 (1955): *Chem. Abs.*, **50**, 2252 (25 Feb. 1956)

Spectrophotometric measurement of the prototropic equilibria between the steps of ionisation of Crystal Violet shows that the mesomeric uni-, bi-, and ter-valent cations are in prototropic equilibrium with one another. C.O.C.

Anhydro Compounds from Nitrogen-containing Derivatives of Thioglycollic Acid. II—Glyoxaline and Benziminazole Compounds—*mero*Cyanine Dyes
G. F. Duffin and J. D. Kendall
J.C.S., 361–368 (Feb. 1956)

(2-Benziminazolylthio)acetic acid (I: R = R′ = X = H) with acetic acid gives a thiazolidone (II), from which *mero*cyanine dyes have been prepared. 1-Substituted acids (I: R′ = X = H), like the corresponding quinoline cpd. (cf. *J.C.S.*, 734 (1951)), give stable anhydro cpd., which may be represented as III, but which, unlike the quinoline anhydro cpd., usually give products derived by attack of the reagent at two different points in the mol.



(I) (II) (III)

H.H.H.

Thermal and Photochemical Air Oxidation of Phenothiazine
G. P. Brown, J. W. Cole, and T. I. Crowell
J. Org. Chem., **20**, 1772–1776 (Dec. 1955)

Water-soluble Sulphur Dyes
H. Boothroyd
Canadian Textile J., **73**, 47–49 (27 Jan. 1956); *Textile J. Australia*, **30**, 1403–1405 (Jan. 1956)

A comparison is made between water-soluble sulphur dyes (Sulphosols) and pseudo-soluble types (mixtures of dye and reducing agents). High solubility and fine particle size are among other properties which make this class superior for many applications to a number of types of fibre. Tendering of cotton by H_2SO_4 degradation is said to be virtually eliminated by the use of Sulphosol Black, which, compared with a normal sulphur black dyed by different methods, yields less H_2SO_4 acidity on dyed cloth after an incubation period of 72 hr. A.H.

Naphthofuroxan and its Derivatives
S. V. Bogdanov and I. N. Korolova

IV—Derivatives of the Dioxime of 1:2-Naphthaquinone-6-sulphonic Acid
J. Gen. Chem. U.S.S.R., **26**, 243–246 (Jan. 1956)

Oxidation of the bisulphite cpd. of 1:2-naphthaquinone-6-sulphonic acid with nitric or nitrous acid gives the bisulphite cpd. of the corresponding naphthofuroxan-sulphonic acid (I), which is converted by Na_2CO_3 soln. into 2-nitro-1-naphthylamine-4:6-disulphonic acid (cf. J.S.D.C., **70**, 308 (1954)). Treatment of I with cold aq. NaOH gives the dioxime of 1:2-naphthaquinone-4:6-disulphonic acid, which is converted by hot aq. NaOH into naphtho[1,2]furazan-5:7-disulphonic acid (cf. J.S.D.C., **70**, 366 (1954)), by acid treatment into 2-nitroso-1-naphthol-4:6-disulphonic acid, and by oxidation into the corresponding naphthofuroxandisulphonic acid.

V—Sulphonation and Chlorination of Naphthofuroxan
Ibid., 264–267

Sulphonation and chlorination of naphthofuroxan occur in position 3 of the naphthalene nucleus, which is therefore probably close in structure to the naphthalene nucleus in the parent naphtho[1,2]furazan, which is substituted in the same position in these reactions. However, the manner of the bonding of the extra oxygen of naphthofuroxan must still be regarded as unsettled (cf. J.S.D.C., **70**, 308 (1954)). A.E.S.

Fluorescent Brightening Agents of the Benzidine Series

I—Synthesis of Benzidine-3:3′-disulphonic Acid
K. Inukai and T. Veda
Rept. Govt. Ind. Research Inst., Nagoya, **1**, 104–106 (1952) *Chem. Abs.*, **50**, 3332 (10 March 1956)

A 66% yield is obtained by treating benzidine with its own weight of 95% H_2SO_4 at 210°C. for 30–35 hr., and separating from the monosulphonic acid by dissolving in dil. acetic acid and adding dil. HCl to precipitate the disulphonic acid.

II—Synthesis of Benzidine-3:3′-disulphonic Acid Derivatives with Various Binding Types
K. Inukai, Y. Maki, and T. Veda
Ibid., 176–182

Synthesis of 23 derivatives, all showing blue to violet fluorescence, is described. C.O.C.

Fluorescent Brightening Agents of the Stilbene Series. I—Influence of Alkali and Sodium Hypochlorite in Preparation of Dinitrostilbenedisulphonic Acid
K. Inukai and Y. Maki
Rept. Govt. Ind. Research Inst., Nagoya, **1**, 98–100 (1952): *Chem. Abs.*, **50**, 3347 (10 March 1956)

An 11–16% yield was obtained by sulphonating 20 g. of *p*-$O_2N·C_6H_4·CH_3$ to give 2:4-$HO_3S(O_2N)C_6H_3·CH_3$. This is treated with 4 c.c. of 35% NaOH and 340–400 c.c. of 5% NaOCl at 50–55°C. Slight excess of NaOCl is beneficial, but the effect of the concn. of the NaOCl is negligible. The product contains the dibenzyl derivative as an impurity. C.O.C.

9:10-Dihydro-9:10-methyleneanthracene
W. R. Vaughan and M. Yoshimine
J. Org. Chem., **21**, 263–264 (Feb. 1956)

2:3-Azadiphenylenetetrazolium Salts and Triazaphenanthrenes
D. Jerchel and H. Fischer
Chem. Ber., **89**, 563–570 (Feb. 1956)

Aromatic Hydrocarbons

LXXI—2:3-4:5-Dibenzoanthanthrene, 4′:1-*o*-Phenylene[naphtho-2′:3′-3:2-pyrene] and 2:3-4:5-Dibenzo[1:12-*o*-phenylene]anthanthrene
E. Clar and W. Willicks
Chem. Ber., **89**, 743–748 (March 1956)

Interaction of 2:3-phthaloylpyrene and 2 mol. of phenylmagnesium bromide yields a diol, readily reduced by KI in acetic acid to 1′:4′-diphenylnaphtho-2′:3′-3:2-pyrene. Brief exposure to an NaCl–$AlCl_3$ melt leads to naphtho-2′:3′-3:2-pyrene and the three cpd. named above. They are obtained by elution from alumina in the order given and their constitutions are deduced from their electronic spectra. 2:3-4:5-Dibenzoanthanthrene (I) yields

(I)

the 6:12-quinone on oxidation with SeO_2 in nitrobenzene. This is a strong red dye for cotton from a violet vat. Its solution in sulphuric acid is green.

LXXII—Relation between Chemical Reactivity, Phosphorescence, and *para*-Absorption Bands, and the "Similarity to Hydrogen" of the Higher Region of the *para*-Bands in the Absorption Spectra of Aromatic Hydrocarbons
E. Clar and M. Zander *Ibid.*, 749–762
H.E.N.

Action of Sulphur Chloride on 2-Aminoanthraquinone
M. K. Bezzubets
J. Gen. Chem. U.S.S.R., **26**, 111–115 (Jan. 1956)

The Herz reaction (preparation of *o*-amino thiols and their deriv. by the action of S_2Cl_2 on an amine or amine salt and suitable treatment of the product) is extended to the conversion of 2-aminoanthraquinone into (2-amino-1-anthraquinonylthio)acetic acid (I), which is diazotised and coupled with various components with formation of azo dyes. Unlike analogous products obtained from aniline and naphthylamines, I does not form a lactam in presence of acids. In previous, unsuccessful attempts to synthesise I, 2-aminoanthraquinone was used in the form of the free base; it is here applied as its hydrochloride in glacial acetic acid. A.E.S.

Derivatives of 4-Hydroxy-2-methylanthraquinone
M. Sulc and J. V. Koštíř
Chem. Listy, **49**, 219–221 (1955): *Chem. Abs.*, **50**, 1719 (10 Feb. 1956)

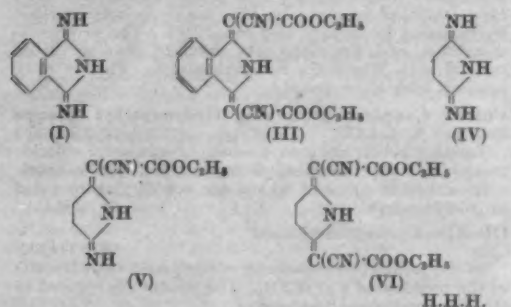
Condensing o-$C_6H_4(CO)_2O$ with *oo'*-dimethyl-*pp'*-dihydroxyldiphenyl gave 4:4'-dihydroxy-2:2'-dimethyl-1:1'-dianthraquinonyl, which, treated over Cu, gave 4:4'-dihydroxy-2:2'-dimethyl*meso*benzodianthrone, and this, treated over $AlCl_3$, yielded the red dye 4:4'-dihydroxy-2:2'-dimethyl*meso*naphthodianthrone. C.O.C.

Synthesis of 1-Acetyl-4-formyl-, 4-Cyanomethyl-, and 4-Carboxymethyl-indole from 1-Naphthylamine
H. Plieninger and K. Suhr
Chem. Ber., **89**, 270–278 (Feb. 1956)

Reactions with Nitrosodisulphonates. XI—Indole and Tetrahydrocarbazole
H.-J. Teuber and G. Staiger
Chem. Ber., **89**, 489–504 (Feb. 1956)

Heterocyclic Imines and Amines. VI—Condensation Products from Di-imino*iso*indoline and Succinimidine with Cyanoacetates
J. A. Elvidge, J. S. Fitt, and R. P. Linstead
J.C.S., 235–243 (Feb. 1956)

Di-imino*iso*indoline (I) condenses with ethyl cyanoacetate (II) (2 mol.) with elimination of NH_3 (2 mol.) to give the *iso*indoline (III); succinimidine (IV) condenses with II (1 mol.) to yield the pyrrolidine (V), but gives the dicondensation product (VI) in the presence of $NaOC_2H_5$. The degradations and ozonolyses of these products are described, their light absorptions are given, and their unexpected properties, e.g. difficulty of clean oxidation and resistance to hydrogenation, are discussed. The light-absorption characteristics support the idea that the formal unsaturation and the functional groups are involved together in mesomerism, since no one of the possible classical bond structures can account for the observed high-intensity maxima.



H.H.H.

3:5-Dichloroaniline in Sandmeyer's Isatin Synthesis. 4:6-Dichloroanthranilic Acid
F. E. Sheibley and J. S. McNulty
J. Org. Chem., **21**, 171–173 (Feb. 1956)

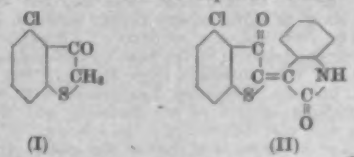
A by-product in the prepn. of the intermediate *α-iso*nitroso-3:5-dichloroacetanilide is *NN'*-bis-3:5-dichlorophenyl*iso*nitrosoacetamidine, which is readily cyclised to 3':4:5':6-tetrachloroisatin *α*-anil. Oxidation of 4:6-dichloroisatin with alkaline H_2O_2 gives 4:6-dichloroanthranilic acid in 83% yield. H.E.N.

Separation of Isomeric Isatins
P. W. Sadler
J. Org. Chem., **21**, 169–170 (Feb. 1956)

A range of *meta*-substituted *iso*nitrosoacetanilides are cyclised in hot sulphuric acid to give, in varying proportions, mixtures of 4- and 6-substituted isatins. Separation of these is best effected by treatment of aqueous soln. of their sodium salts with glacial acetic acid, which precipitates the 4-isomers. The others are obtained on addition of HCl to the mother-liquors. H.E.N.

Indigoid Vat Dyes of the Isatin Series. IX—Dyes from 4-Chloro-3-hydroxythionaphthen
S. K. Guha, J. N. Chatterjee, and A. K. Sinha
J. Indian Chem. Soc., **32**, 777–781 (Dec. 1955)

2-Mercapto-6-chlorobenzoic acid has been prepared from the corresponding anthranilic acid, which was obtained by oxidation and subsequent reduction of 2-chloro-6-nitrotoluene, by diazotising and reaction with potassium ethyl xanthate. This with chloroacetic acid followed by cyclisation gave 4-chloro-3-hydroxythionaphthen (I), which has been condensed with isatin and its halogeno and nitro derivatives to give the corresponding thioindigoid dyes (II) and oxidised with potassium ferricyanide to 4:4'-dichlorothioindigo. These dyes gave reds and violet–reds on cotton which were hypsochromic in comparison with those from the corresponding 6-chloro compound but bathochromic with respect to those from the 5- and 7-chloro derivatives reported earlier.



C.H.R.

The Iron(II)–Methin Chromophore
D. H. Busch and J. C. Bailar
J. Amer. Chem. Soc., **78**, 1137–1142 (20 March 1956)

The intensely coloured complexes formed by iron(II) with heterocyclic diamines and triamines and with *α*-di-imines form a single class of compounds. A structural link between the complexes of aromatic and heterocyclic diamines and *α*-di-imines is found in the iron(II) derivative of pyridinalmethylimine, which has both structural units and physical properties in common with those of the two extreme types of ligand. From the magnetic moments and visible, ultraviolet, and infrared spectra of the iron compounds of this class it is deduced that the behaviour and the properties of these compounds are best described in terms of six σ-bonds formed between the metal and the six methin N atoms, the strengths of which depend on the basicity of the ligand, and three π-bonds resulting from interaction of the unhybridised *d*-electron pairs of the metal atom with the π-system of the ligand. The strength of the π-bonds varies with the degree of conjugation in the chelate ring. C.O.C.

Infrared Spectra of Phthalocyanines and the Effects of Gaseous Substances
A. N. Sidorov and A. N. Terenin
Doklady Akad. Nauk S.S.S.R., **104**, 575–578 (1 Oct. 1955)

Films (0·01 mm. in thickness) of phthalocyanine and its Fe, Co, Mg, Zn, and Cu deriv. are prepared on sylvine plates by sublimation *in vacuo*, and their infrared absorption spectra are determined. Water (ordinary or heavy), H_2S, hydrazine, phenylhydrazine, NH_3, benzylamine, aniline, diphenylamine, pyridine, or O_2 is then admitted as gas or vapour at low pressure, and after 15–20 hr. the infrared spectra are redetermined. Interaction with the vapour is indicated by substantial changes in the spectra, both of the dye and of the addend. Definite interaction

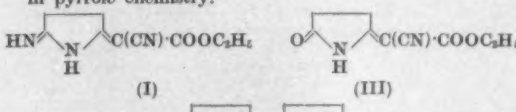
is observed only for the Mg, Zn, and Fe deriv., phthalocyanine itself and its Cu deriv. being unaffected. There is no interaction with H_2S, NH_3, O_2, or diphenylamine. A.E.S.

Colorimetric Measurements on Phthalocyanine Pigments
G. Ghisolfi
Ind. vernice (Milan), **9**, 181–183, 203–205 (1955): *Chem. Abs.*, **50**, 2987 (25 Feb. 1956)

Colour evaluations by the tristimulus method were made of the following copper phthalocyanine pigments: (*a*) unstable commercial pigment prepared by the acid-pasting method; (*b*) 10 g. of (*a*) heated to 480°c. for 30–35 min. in a covered crucible in a muffle furnace in an inert atmosphere; (*c*) solvent-stable pigment obtained by grinding 30 g. crude pigment with 300 g. NaCl and 100 g. 1:1 xylene–toluene for 48 hr. in a small iron mill using steel balls, filtering, vacuum-drying, washing with ether and then ethanol, drying, treating with 2% H_2SO_4 at 60°c. for 1 hr., filtering, washing free from SO_4 ions with water, and drying; (*d*) 10 g. pigment in 100 ml. 1:1 xylene–toluene heated at 50°c. for 48 hr., filtered, washed with ether and then ethanol, and dried. Effect of solvent treatment is similar with both the stable and the unstable pigments, and both types of pigments were similar before and after extraction; thus the difference in colour between the stable and unstable pigments is caused by differences in aggregation or flocculation and not in basic hue. The effect of solvent extraction was most marked with the less thoroughly ground unstable pigment, the reverse being true for the stable pigment. Heat-treated pigment was not affected by solvents. Change of tinting strength with grinding was greater with the unstable pigment. Solvent extraction lowered the tinting strength of the unstable pigment by 17·5% (after the tenth extraction), but that of the stable pigment fell only by 9%. When the pigment had been heat-treated, both the solvent-extracted and unextracted pigments showed progressive increases in tinting strength as grinding went on, the extracted pigment showing 2·5% greater tinting strength than the unstable untreated pigment after six runs through the mill. The colour components of the heat-treated pigment were at the start very different from those of the original pigment, but the differences disappeared with grinding. The ultraviolet spectra of the unstable and the crude pigments were very different, but the spectrum of the heat-treated pigment resembled that of the stable (*a*) form. C.O.C.

Compounds containing Directly Linked Pyrrole Rings. I— New Type of Pyrrole Pigment
J. A. Elvidge, J. S. Fitt, and R. P. Linstead
J.C.S., 244–246 (Feb. 1956)

A new magenta pigment, di-(5-cyanoethoxycarbonylmethylene-2-pyrrolidinylidene) (II), is formed in low yield when the iminopyrrolidine (I) is boiled in propionic acid and the soln. kept at room temp. Oxidation of II with $KMnO_4$ in aq. H_2SO_4–acetic acid gives the pyrrolidone (III). The colour and the fine structure of II and its relation to indigo are discussed. The finding that an intensely coloured cpd. can result from the direct linking of two partly pyrrolic rings as in II seems to be a new discovery in pyrrole chemistry.



H.H.H.

Chlorophyll—Chemistry, Manufacture, and Applications
J. M. de Palacio MacLennan
Afinidad, **32**, 188–193 (Sept.–Oct. 1955)

A brief review. S.R.C.

Review of Anthraquinone Compounds in Lichens
S. Subramanian
J. Amer. Pharm. Assocn., **44**, 769–776 (1955): *Chem. Abs.*, **50**, 3712 (10 March 1956)

23 references. C.O.C.

Chemistry of *Damnacanthus* Genus
S. Nonomura

I— Anthraquinone Colouring Matter from *D. major*, *D. major var. pavifolius*, and *D. indicus var. microphyllus*—1 and 2
J. Pharm. Soc. Japan, **75**, 219–224 (1955)

The following pigments were isolated from the roots—damnacanthal, juzunal, damnacanthole, damnidin, and a neutral compound. Juzunal is shown to be hydroxydamnacanthal, and damnacanthole to be lucidin *a*-methyl ether.

II— Constitution of Damnacanthal
Ibid., 225–227

Damnacanthal is 1-methoxy-2-formyl-3-hydroxyanthraquinone. C.O.C.

Di- and Poly-cyclic Azulenes. XXIII— Synthesis of Azulenes by Ring Expansion with Nascent Aliphatic Diazo Compounds
W. Treibs, M. Quarg, and E. J. Poppe
Annalen, **598**, 32–37 (March 1956)

Anthocyanin Pigments of Sour Cherries
C. L. Kuang and A. C. Wagenknecht
J. Amer. Chem. Soc., **78**, 979–980 (5 March 1956)

Galloflavin. III— Position of the Carboxyl Group in *iso*Galloflavin and a Synthesis of Trimethylbreviflolin
J. Grimshaw and R. D. Haworth
J.C.S., 418–422 (Feb. 1956)

Total Synthesis of Perhydrocrocetin and Preparation of Moieties of Carotenoid Structure
E. Buchta and H. Schlesinger
Annalen, **598**, 1–24 (March 1956)

Carotenoid Syntheses. XXXIV— Preparation of C_{14}- and C_{15}-Aldehydes
H. H. Inhoffen and D. Erdmann
Annalen, **598**, 51–64 (March 1956)

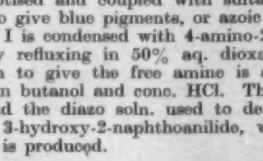
Titanium Dioxide from Bauxite Sludge
V. Damodoran and J. Gupta
J. Sci. Ind. Research (*India*), **14 B**, 292–297 (1955): *Chem. Abs.*, **50**, 3775 (10 March 1956)

TiO_2 prepared by normal methods from bauxite sludge is an inferior pigment because of the large amount of Al_2O_3 in the sludge. A method of removing the Al_2O_3 before pigment formation is described. C.O.C.

PATENTS

Derivatives of *s*-Triazine as Diazo Components for Azoic Blues
American Cyanamid Co. *USP* 2,688,616

Condensation of 6-chloro-2:4-diphenyl-*s*-triazine (1) with 4-amino-2:5-dialkoxyacylanilides gives compounds of formula—



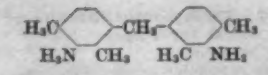
Hydrolysis of the acyl group then gives amines, which may be diazotised and coupled with suitable coupling components to give blue pigments, or azoic dyeings and prints. Thus I is condensed with 4-amino-2:5-diethoxyacetanilide by refluxing in 50% aq. dioxan and HCl. De-acetylation to give the free amine is accomplished by refluxing in butanol and conc. HCl. The product is diazotised, and the diazo soln. used to develop cotton padded with 3-hydroxy-2-naphthoanilide, when a very greenish blue is produced. E.S.

Colour Couplers containing Hydroxyalkyl Groups
Eastman Kodak Co. *USP* 2,710,803

Treating colour couplers containing sulphonyl chloride groups with hydroxyl-containing amines, e.g. ethanolamines, yields couplers which are readily incorporated in gelatin emulsions. C.O.C.

Diaminodiphenylmethanes
Ciba *BP* 744,303

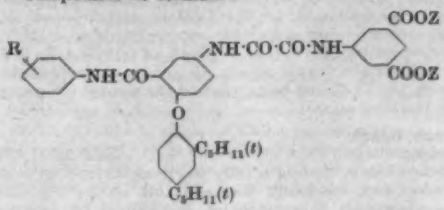
2:6-Dimethyl-1-nitrobenzene is condensed with H·CHO in the presence of e.g. H_2SO_4. The product is reduced to the diaminodiphenylmethane—



useful as a disazo dye component. R.K.F.

Dialkyl 5-Oxanilamido*iso*phthalate Colour Couplers
Eastman Kodak Co. *USP* 2,710,802

Compounds of formula—



(R = a coupling group in *meta* or *para* position; Z = Alk or an alkali metal), e.g. dimethyl 5-chloroglyoxylamido-*iso*phthalate, are colour couplers which are readily purified by recrystallisation and are readily incorporated in gelatin emulsions. C.O.C.

Hydroxyindazolesulphonic Acids
ICI *BP* 744,212

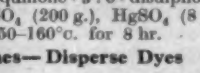
An aminoindazolesulphonic acid is heated with water or dil. mineral acid under pressure to produce the corresponding hydroxyindazolesulphonic acid. Thus Na 6-aminoindazole-4-sulphonate is heated at 170–175°C. for 10 hr. with ca. 10% aq. H_2SO_4. The H_2SO_4 is removed as $CaSO_4$, and the filtrate neutralised with $NaHCO_3$ and evaporated to dryness. R.K.F.

Chloroanthraquinonetrisulphonic Acids
FBy *BP* 744,395

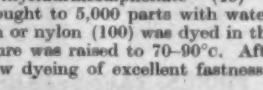
2-Chloranthraquinone-3:7- and -3:6-disulphonic acids are further sulphonated with excess of SO_3 in presence of <4% Hg salt on wt. of disulphonic acid to give respectively the 3:5:7- and the 3:6:8-trisulphonic acid. Thus Na 2-chloroanthraquinone-3:6-disulphonate (100 g.) is heated with H_2SO_4 (200 g.), $HgSO_4$ (8 g.), and 65% oleum (200 g.) at 150–160°C. for 8 hr. R.K.F.

Nitroketoarylamines— Disperse Dyes
General Aniline *USP* 2,708,149

Compounds of formula—



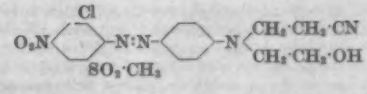
(R = Alk of 1–5 C; X = a carbocyclic or a heterocyclic radical) are greenish-yellow to reddish-yellow to brown disperse dyes of outstanding fastness properties, particularly to light. Thus 3-nitro-4-chloroacetophenone (6 parts), aniline (2·79), $NaHCO_3$ (2·5), and water (50) were refluxed for 16 hr. The viscous oil formed on cooling was separated from the water and crystallised from *iso*propyl alcohol to give an 84% yield of the yellow dye—



This dye (1–3) was dissolved in a small amount of a 50:50 mixture of alcohol and acetone, 5% aq. sodium *N*-oleyl-*N*-methyltaurinesulphonate (40) added, and the whole brought to 5,000 parts with water at 45–55°C. Acetate rayon or nylon (100) was dyed in this bath while the temperature was raised to 70–90°C. After soaping, a greenish-yellow dyeing of excellent fastness to light was obtained. C.O.C.

Brown Monoazo Disperse Dyes
S *BP* 746,475

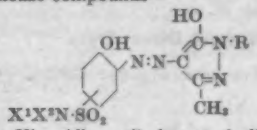
Monoazo compounds formed by diazotising 6-chloro-4-nitro-2-alkylsulphonylanilines and coupling with *N*-cyanoalkyl-*N*-alkyl (or -hydroxyalkyl) derivatives of aniline, toluidine, etc. are brown disperse dyes for cellulose acetate, nylon, and polyester fibres. Thus 6-chloro-4-nitro-2-methylsulphonylaniline is diazotised in nitrosylsulphuric acid and coupled with *N*-cyanoethyl-*N*-hydroxyethylaniline to give—



E.S.

Metal(Cobalt and Chromium)-complex Monoazo Dyes for Wool and Nylon
Ciba *BP* 746,944

The Co and Cr complexes containing 1 atom of metal to 2 mol. of monoazo compounds—



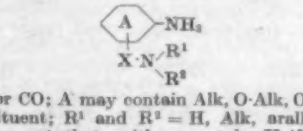
(R = Alk or Ar; X^1 = Alk, aralkyl, or *cyclo*alkyl; X^2 = H or Alk; or X^1 and X^2 together form a heterocyclic residue with N), or to 1 mol. of such a monoazo compound and 1 mol. of an *oo'*-dihydroxymonoazo compound of the phenylazonaphthalene series free from SO_3H and COOH groups, dye wool and nylon from weakly acid, neutral, or weakly alkaline baths. Thus, 2-aminophenol-5-sulphon-*N*-methylamide is diazotised and coupled with 1-*n*-butyl-3-methyl-5-pyrazolone. Boiling with aq. sodium chromosalicylate converts it into the Cr complex, which dyes wool orange from a neutral or an acetic-acid bath. E.S.

Metal(Chromium)-complex Monoazo and Monoazomethin Dyes for Wool, Nylon, etc.
BASF *BP* 747,367

Water-soluble chromium complexes, which dye wool, nylon, etc. from neutral or weakly acid baths, are made by treating metal complexes containing 1 atom of Cr to 1 mol. of suitable monoazo or monoazomethin compounds with colourless aromatic chelating compounds, e.g. salicylic acid. The monoazo and monoazomethin compounds used contain one metallisable grouping, at least one sulphamyl group and/or one amino or acylamino group, any free amino group being acylated after formation of the mixed chromium complex, and no SO_3H group. Thus the chromium complex containing 1 Cr to 1 mol. of the monoazo compound 2-amino-5-nitrophenol→2-naphthol-6-sulphonamide is boiled for 2 hr. with aq. sodium acetate and the 5-sulphonanilide of salicylic acid. The resulting complex dyes wool reddish blue from a weakly acid bath, and levels better than the similar dye prepared from 2-amino-5-nitrophenol and β-naphthol. E.S.

Blue Metallisable Disazo Direct Dyes
Ciba *BP* 746,877

Direct dyes which may be metallised with nickel, or preferably with copper, compounds, in substance, in the dyebath, or on the fibre, are made by coupling tetrazotised 3:3'-dihydroxybenzidine (I) with 2 mol. of an *N*-phenyl-J acid or *N*-phenyl-γ acid having in the *N*-phenyl group a sulphamyl or substituted sulphamyl group. Thus J acid is boiled with metanilamide in presence of sodium sulphite to give—



Two mol. of this coupling component are coupled with 1 mol. of tetrazotised I in presence of $Ca(OH)_2$ and pyridine to give the disazo dye, which dyes cotton greenish blue by the single- or two-bath aftercoppering process. E.S.

Brown Azoic Dyes for Cellulose Acetate, Nylon, etc.
FH *BP* 747,487

The components of the monoazo pigments described in *BP* 511,432 and 512,864 (*J.S.D.C.*, **56**, 40 and 84 (1940)), applied to cellulose acetate, nylon, etc. by the azoic process, give browns fast to cross-dyeing. The diazo components have the formula—

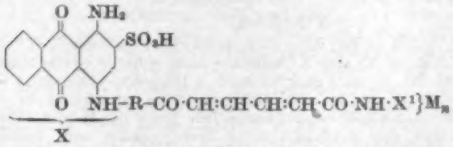
(X = SO_2 or CO; A may contain Alk, O·Alk, O·Ar, or Hal as a substituent; R^1 and R^2 = H, Alk, aralkyl, Ar, or *cyclo*alkyl, except that neither must be H if the group

$X{\cdot}NR^1R^2$ is *ortho* to the NH_2 group; in addition R^1 and R^2 may together form a heterocyclic ring with the N atom), and the coupling components are arylides of 2-hydroxycarbazole-3-carboxylic acid (I) or of 2-carboxy-3-hydroxydiphenylene oxide or sulphide. Thus cellulose acetate is worked at 75°C. in a bath containing diethanolamine, ammonia, the Na salt of the *p*-chloroanilide of I, and 2:5-dimethoxyaniline-4-sulphondi-*n*-butylamide. Diazotisation is then carried out in a cold nitrous-acid bath, and the dark brown dyeing developed by treatment in a slightly alkaline bath at 60°C. E.S.

Anthraquinonoid Direct Dyes

BASF *BP* 744,441

Green substantive dyes for cellulose of formula—



(R = phenylene or diphenylene; X^1 = X, or similarly substituted anthraquinone residue, or residue of yellow azo dye; M = "heavy" metal; *n* = 0–2; the $-CH{:}CH{\cdot}CH{:}CH-$ may contain CH_3 or Cl; the 5, 6, 7, or 8 position of the anthraquinone nucleus may carry SO_3H, and 6 and 7 one or two halogen atoms) are made e.g. by running 1:3-butadiene-1:4-dicarboxylic chloride dissolved in CCl_4 into aq. 1-amino-4-*p*-aminophenylaminoanthraquinone-2:6-disulphonic acid containing NaOH at 0–5°C. After removal of the CCl_4, the solution is acidified, and the dye filtered off, neutralised with Na_2CO_3, and dried. Suitable dyes are metallised by treatment with Cu or Ni salts in presence of ammonia or an organic base. R.K.F.

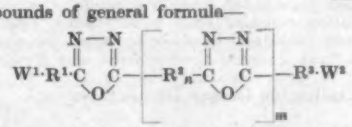
Fluorescent Brightening Agent

Ciba *BP* 746,046

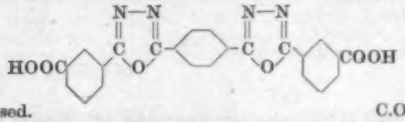
Compounds containing one or more 1:3:4-oxadiazole rings exhibit green–blue to violet fluorescence in ultraviolet radiation. They may be used on various substrates depending on the affinity conferred by the rest of the molecule.

BP 746,047

Compounds of general formula—



(R^1 and R^3 = aromatic, aliphatic, or araliphatic radicals; R^2 = aliphatic or aromatic radical; at least one of R^1, R^3, and R^3 contains a system of conjugated double bonds which are in conjugated relation with the double bonds of the adjoining oxadiazole ring; W^1 and W^2 = sulpho, carboxyl, or amino; *m* = 1 or 2; *n* = 0 or 1), e.g.—

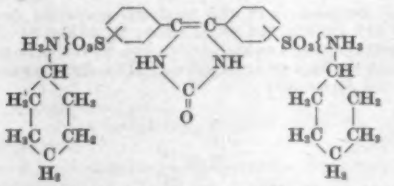


are used. C.O.C.

Fluorescent Brightening Agent for Vinyl-type Plastics, etc.

General Aniline *USP* 2,709,702

The amine salts of 4:5-diphenyliminazolonedisulphonic acids, e.g.—



are spirit-soluble and so can be readily incorporated in lacquers, varnishes, vinyl-type plastics, etc. They fluoresce blue under ultraviolet radiation. C.O.C.

α - Benzyl - β - methylumbelliferone — Fluorescent Brightening Agent for Paper

Davies-Young Soap Co. *USP* 2,710,285

This agent is applicable in wax coatings. C.O.C.

Chlorinated Copper Phthalocyanine Pigments

DuP *BP* 745,772

A mixture of (*a*) cupric chloride, (*b*) phthalic acid or anhydride, mono- or di-ammonium phthalate, phthalamide, or phthalimide, (*c*) the 4-chloro derivative of (*b*), and (*d*) urea is heated in an inert organic liquid at $<190°C$. The molar ratio of (*a*) : (*d*) is 1 : 15–40, of (*c*) : (*b*) 1–3 : 3–1. The products are copper mono-, di-, or tri-chlorophthalocyanines, the Cl atoms being *meta* to the nearest CN group in the benzene nuclei. C.O.C.

Furnace Black

Columbian Carbon Co. *USP* 2,707,672

Furnace black, having a fully oxidised surface, intense blackness, and markedly increased oil absorption and iodine adsorption characteristics, is obained by heating the pigment to >650°F. but not to red heat in a current of air or of air and steam.

USP 2,707,673

The product obtained by the above process is heated at 1800–2000°F. in a non-oxidising atmosphere. This gives a black of greatly increased iodine value.

USP 2,707,674

Furnace blacks may be oxidised so as to develop pH as low as 2·5, with no appreciable loss in weight or reduction in particle size, by treating them by the process of *USP* 2,707,672 (above). C.O.C.

Amine-coated Calcium Carbonate Pigments

Wyandotte Chemicals Corpn. *USP* 2,709,160

Calcium carbonate is readily incorporated in rubber stock if the pigment has been coated with a primary *n*-alkylamine (Alk of 10–18 C) or rosin-amine. C.O.C.

Zinc Sulphide stabilised to Light

Monsanto *USP* 2,711,968

A pigment which is not discoloured by exposure to actinic radiation consists of ZnS (100 parts by wt.) and MgO of particle size 0·05–0·1 μ. (40–45). C.O.C.

Colloidally Dispersed Oxides for the Textile Industry (III p. 294)

Dyeing Wool, Nylon, etc. with Metal(Cobalt)-complex Monoazo Dyes from Baths containing Phosphates (VIII p. 306)

Metal-complex Azo Dyes as Printing Assistants in the Application of Thioindigoid Vat Dyes (IX p. 306)

Colour Photography—Styryl Dyes as Dischargeable Yellow Filters (IX p. 307)

V— PAINTS; ENAMELS; INKS

PATENT

Two-coloured, Wallpaper-like Surface Coatings

J. Spillmann-Zuppinger and A. Jud *BP* 745,916

A coating material contains two differently coloured immiscible phases, one of them being aqueous and the other non-aqueous. At least the aqueous phase contains cellulosic material. When the coating is applied by any means, the non-aqueous phase uniformly covers the primed ground, while the aqueous phase separates, so that the cellulosic fibres in it are deposited on the surface. On drying, the coating looks like wallpaper. C.O.C.

VI— FIBRES; YARNS; FABRICS

Dielectric Properties of Fibre Assemblies

J. W. S. Hearle

Text. Research J., **26**, 108–111 (Feb. 1956)

Previous work on the dielectric properties of a wide range of textile fibres in yarn form (cf. J.S.D.C., **70**, 466 (1954)) has been extended to cover the frequency range 10 kc./sec. to 10 Mc./sec. Effective dielectric constants and dissipation factors for the air–fibre mixtures are given, and the effects of frequency and moisture content are discussed. J.C.F.

Influence of Ultraviolet Rays on the Tensile Strength of Cotton Fabrics

F. Bathorij *et al.*

Magyar Textiltech., (7), 231–232 (1954): *Hungarian Tech. Abs.*, **7**, (2), 19 (1955): *J. Textile Inst.*, **47**, A 50 (Jan. 1956)

Cotton fabrics on ultraviolet irradiation suffer damage owing to transformation of cellulose into photocellulose, whose properties are similar to those of oxycellulose. The region of the destructive rays is within the quartz ultraviolet range (wavelength $< 350\,m\mu$). Twilled cotton fabric, irradiated by solar rays for 700 hr. (total radiating energy of 35,696 cal./min. per sq.cm.), showed a tensile strength loss of 46·5% in the warp direction and of only 10% in the weft direction. This difference was due to the weave, in which most of the warp yarns were on the surface. Dyed cloths are less susceptible to damage; when irradiated under similar conditions, the tensile strength decreased only by 19·1% in the warp direction. C.J.W.H.

Nature of Structural Viscosity and the Properties of Concentrated Viscose Solutions

N. V. Mikhailov and N. N. Zav'yalova

J. Appl. Chem. U.S.S.R., **29**, 97–105 (Jan. 1956)

Lateral Order Distribution in Cellulose Fibres

H. Maeda

I— Method of Measuring the Lateral Order Distribution in Regenerated Cellulose Fibres

J. Soc. Textile Cellulose Ind. Japan, **12**, 6–10 (Jan. 1956)

When heated in an alcoholic solution of HCl, cellulose fibres could be depolymerised without undergoing such vigorous crystallisation as in the case of hydrolysis in aqueous acid. Thus pretreated samples were subjected to solubility determinations in NaOH soln. of various concn., and summative mass-order relations could be obtained by plotting the amounts dissolved against the concn. of soln. in which the solubilities were measured. By differentiating the mass-order curve, the lateral order distribution for the original sample was obtained, similarly to the case of determination of D.P. distribution of cellulose materials. The lateral order distribution curve for viscose rayon staple fibre indicated two peaks, probably corresponding to the existence of skin–core heterogeneous structure in fibre cross-section. Standard conditions of measurement were— predepolymerising bath a mixture of 5 vol. of 3·5 N-HCl (aq.) and 95 vol. of absolute alcohol, temp. of bath 80°C., duration of depolymerisation 6 hr., temp. at which the solubilities in NaOH were measured 25°C.

II— Lateral Order Distribution of Several High-tenacity Rayons

Ibid., 10–13

Using the method described above, the lateral order distributions of four domestic high-tenacity rayons were investigated. They also have two peaks in the distribution curve, and the greater the amount distributed in the lower-order region, the better the mechanical properties, especially the durability or resistance to fatigue. The excellent mechanical properties of super high-tenacity rayons such as Super Cordura seem likely to originate from a lower lateral order combined with the homogeneous structure of the fibre cross-section. In contrast to these rayons, the lateral order distribution curves of regenerated cellulose fibres of another group, including Fortisan, Lilienfeld rayon, BX Fibre, Toramomen, etc., give only one peak in the region of very high lateral order. AUTHOR

Extensometric and Elastic Properties of Textile Fibres

B. Farrow

J. Textile Inst., **47**, T 58–T 101 (Feb. 1956)

Using a Cambridge textile extensometer, the elastic properties of approx. 60 fibre samples, representing 15 chemical classes of man-made fibres with some natural fibres for comparison, are studied. They are stretched air-dry and in cold and hot water, and the effect of hot-water pretreatment is also investigated. Results are tabulated, and illustrated by graphs, in detail. J.W.B.

Degree of Cellulose Polymerisation in Ramie Fibre

S. Lewandowski and Z. Gotwald-Orlowska

Prace Inst. Wlok., **4**, (17), 22–29 (1955): *J. Textile Inst.*, **47**, A 47 (Jan. 1956)

Although it is known from literature data that the ramie fibre has the highest D.P. compared with other cellulosic fibres, none of the publications disclose the D.P. in ramie from different sources and in various parts of the stem, especially in connection with processing properties and the usefulness of these fibres. The investigations described were carried out on Chinese ramie samples; the results show that the fibres with best processing properties are found in the upper part of the stem. These fibres have a lower D.P. than those from other parts of the plant, and are, therefore, less rigid. C.J.W.H.

Surface Friction of Fabrics

Y. Miura

II— Slippage of Worsted and Woollen Fabrics

J. Soc. Textile Cellulose Ind. Japan, **12**, 41–44 (Jan. 1956)

III— Kinetic Friction of Fabrics

Ibid., 44–47

Reactions of Aromatic Fluoro Compounds with Amino Acids and Proteins. XV— Reaction of 4:4′-Difluoro-3:3′-dinitrodiphenyl Sulphone with Fibroin

H. Zahn, H. Zuber, W. Ditscher, D. Wegerle, and J. Meienhofer

Chem. Ber., **89**, 407–414 (Feb. 1956)

30% of the tyrosine of fibroin is left unreacted. The increase in wt. shows that all of the reagent acted bifunctionally with Italian silk, but only 75% of it acted thus with Japanese silk. The reaction occurs to about 70% with the tyrosine moiety, but also with lysine to a small extent. That pairs of ε-amino groups of the lysine residues can be covalently linked by such a bifunctional reagent shows that they must lie close together, which accounts also for their quantitative interaction with polybasic acids. H.E.N.

Light Transmission and Air Flow through Nylon Parachute Fabrics

A. Baker and J. E. Swallow

J. Textile Inst., **47**, T 102–T 109 (Feb. 1956)

Measurements are made of light transmission, air flow, and bubble pressure on a series of dyed and undyed, plain- and twill-weave, nylon parachute fabrics. Results indicate a characteristic linear dependence of air flow on open area for each fabric examined, and variations of open area in a given fabric are associated with spreading of filaments, especially in the weft. J.W.B.

Structure of Synthetic Polyamide Fibres. VI— Integral Heat of Solution of Capron [6-Nylon] Fibre in Formic Acid

N. V. Mikhailov and É. Z. Fainberg

Kolloid. zhur., **18**, 44–48 (Jan.–Feb. 1956)

Determinations are made of the integral heat of soln. of Capron in 95% formic acid (final concn. 1–2% of Capron) and the heat of dilution of a 15% soln. of Capron in 95% formic acid. The heat of soln. is 12·6 cal./g. for undrawn fibre and 11·5 cal./g. for cold-drawn fibre (250% draft); the difference may arise from elimination of crystal defects and/or changes in packing of crystallites during drawing. Unlike soln. of cellulose deriv., for which the heat of dilution is almost zero, a 15% soln. of Capron has a finite positive heat of dilution, substantially const. at ~1·4 cal./g. over a range of final concn. of 0·5–2·5%; this fact indicates that the heat of solvation exceeds the energy required to separate the chains. A.E.S.

Ageing of Silon (6-Nylon) Fibres

L. Urbancova

Chem. Průmysl, **5** (30), (2), 78–80 (1955): *Chem. Abs.*, **49**, 12010 (10 Sept. 1955): *J. Textile Inst.*, **47**, A 49 (Jan. 1956)

Comparative results of accelerated ageing tests in a German weatherometer and of outside roof exposure of Silon caprolactam fibres (equivalent to Perlon L) are evaluated. The degradation is a thermal and photochemical depolymerisation resulting in decreased mechanical strength, increased brittleness, and change of colour. If not exposed to the extreme effects of light and heat, and at the normal moisture content, the fibres do not show appreciable degradation. C.J.W.H.

Enant— A New Polyamide Fibre

A. A. Strepikheev, V. N. Topchibasheva, and T. I. Shein
Tekstil. prom., **16**, 33–34 (Jan. 1956)

The new fibre Enant (7-nylon) is to be preferred to Capron (6-nylon) in that it contains no free monomer, is more thermostable (contents of benzene- and water-sol. components and viscosities of melt and cresol soln. remain unchanged after 12 hr. at 260°c.), and suffers less degradation when exposed to ultraviolet radiation. In its other properties—mechanical, physical, and chemical—Enant is very close to Capron. The materials required for the manufacture of the fibre are now readily available.
A.E.S.

PATENTS

Acrylonitrile Polymers having Good Affinity for Acid Dyes

FBy *BP* 745,687

Introduction into acrylonitrile polymers of polycondensates containing in the main carbon chain N atoms more than two of whose valences are linked to C atoms, e.g. the basic polyurethan obtained from hexamethylene di-*iso*cyanate and methyl-diethanolamine, imparts increased affinity for acid dyes.
C.O.C.

Sized Glass Filaments

Owens-Corning Fiberglas Corpn. *USP* 2,712,509

The chemical and heat resistance of glass fibres as well as their physical and electrical properties are improved by coating them with a dispersion of polytetrafluoroethylene and simultaneously or subsequently with a fugitive binder, e.g. a wax or starch, which is removed on heating to 650–900°c. to leave a uniform coating of polytetrafluroethylene on the fibres.
C.O.C.

Warp-pile Fabrics (VIII p. 305)
Characteristics of "Wash-and-wear" Cottons (X p. 308)
Chemical Bases of the Felting and Milling of Protein Fibres (X p. 309)
Heterogeneous Hydrolysis of Cotton and Sulphite Cellulose (XI p. 310)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Launderable Textiles in 1955

G. H. Johnson
Amer. Dyestuff Rep., **45**, P 122–P 128 (27 Feb. 1956)

Reagents and recipes used in the laundering trade are surveyed (with detailed examples), and current problems discussed, together with methods of studying them. The survey is systematic, fabrics made from natural and man-made fibres being discussed in turn with regard to their behaviour in laundering.
J.W.B.

Colour and Finishing Problems in Dry Cleaning

J. Parsley
Amer. Dyestuff Rep., **45**, P 128–P 130 (27 Feb. 1956)

Problems arising from consumer complaints relating to colour faults in dry cleaning are briefly surveyed. These include imperfect penetration, leading to "crowsfeet" (Monastral Blue is quoted), dye solubility in standard cleaning reagents, resin-bonded dyes, faulty colour choice, topping, heat-sensitive dyes which break down on pressing (e.g. light-weight acetate–viscose rayon trousers), and non-permanent finishes.
J.W.B.

Continuous Preparation and Bleaching of Rayon Staple Fabrics

M. P. Kozlov
Tekstil. prom., **16**, 40–42 (Jan. 1956)

Loom-state rayon staple fabric is padded at 40–45°c. in a soln. containing, per litre— 3–4 g. of H_2O_2, 6–7 g. of sodium silicate soln. (sp.gr. 1·4), 1–1·5 g. of NaOH, and 0·5 g. of Wetting Agent OP–10; the pick-up is 100–120%. The fabric is then steamed for 15 min. and washed with hot and cold water. Under these conditions satisfactory desizing and bleaching occur without degradation.
A.E.S.

PATENT

Bleaching Keratinous Fibres

Harris Research Laboratories *BP* 745,941

The fibres, preferably after an alkaline scour, are treated with an aq. soln. of formaldehyde and a ferrous salt, preferably $FeSO_4$, at 65–100°c. and pH 2·8–9·5, and are then treated with aqueous peroxide at 40–90°c. and pH 7–9. This enables alpaca, cashmere, etc. to be bleached without affecting strength, softness of handle, or other desirable properties.
C.O.C.

Warp-pile Fabrics (VIII p. 305)
Chemical Bases of the Felting and Milling of Protein Fibres (X p. 309)

VIII— DYEING

High-temperature Dyeing in Circulating-liquor Machines

B. Steverlynck
Textil Praxis, **11**, 155–160 (Feb. 1956)

A description of the Steverlynck system for dyeing textiles at high temp. Reference is made to the danger of decomposition of many direct dyes, the improved exhaustion with sulphur dyes on cellulosic materials, the necessity of not exceeding 105–108°c. in wool dyeing, and the value of the high-temperature dyeing of synthetic fibres.
B.K.

Experiments and Plant Experience with Continuous Dyeing

K. Messik, I. Bass, and A. Szabolcs
Magyar Textiltech., (1), 31–34 (1955): *Hungarian Tech. Abs.*, **7**, (4), 16 (1955)

In the majority of continuous dyeing processes impregnation with dye liquor and fixation are separated in both time and space. The purpose of padding is the even impregnation of fibres. In padding unevennesses in the cross-section may occur owing to the low penetration of dye, one of the causes of which is high interfacial tension between the fibres and the solution. The other is that the concentration of the dye liquor undergoes a reduction in the course of absorption by the fabric and thus the liquor that reaches the interior of the fabric is considerably weaker. The phenomenon called tailing is occasioned by dyeing during the time of padding. All the factors which improve dye penetration also favour the elimination of changes in the depth of colour at the beginning of the padding, i.e. tailing. The relation between the variation of colour and time is given by an exponential equation, the pad trough volume playing a decisive rôle. A minimum affinity is maintained for the elimination of unevenness in padding. Vat dyes are padded in the form of pigment suspensions. The continuous dyeing process using vat pigment, elaborated by the [Hungarian] Research Institute of the Textile Industry, employs Rongalite as a reducing agent. Squeezing and liquid turbulence are applied to improve dye penetration. The hydrostatic pressure of a metal bath also can be utilised for the removal of excess liquid. For heating purposes, some methods use steam, others heated liquids, e.g. a metal bath or an oil bath. The main object of the soaping of vat dyeings is to develop fully fastness values. This frequently cannot be obtained in open-width washers.
C.J.W.H.

Inhibition of Fading by Light— I

Z. Hajós and J. Fodor
Magyar Tudományos Akad. Kém. Tudományok Osztályának Közleményei, **5**, 533–543 (1955): *Chem. Abs.*, **50**, 2176 (10 Feb. 1956)

A similar mechanism is proposed for both autoxidation and fading on exposure to light.
C.O.C.

Garment Dyeing in the United States Today

A. Teichner
Amer. Dyestuff Rep., **45**, P131–P133 (27 Feb. 1956)

Problems of the wholesale garment-dyeing industry are briefly discussed.
J.W.B.

Thermodynamic Investigation of Cotton Dyeing

P. V. Morÿganov and B. N. Mel'nikov
Kolloid. zhur., **18**, 49–60 (Jan.–Feb. 1956)

An account with full experimental details of work already reported briefly (see J.S.D.C., **72**, 187 (April 1956)).
A.E.S.

Dye Accessibility of Rayon and Synthetic Fibres I— Dissociation Energy of the Direct Dye-Fibre Complex

Y. Konishi and K. Shibata
J. Soc. Textile Cellulose Ind. Japan, **12**, 48–53 (Jan. 1956)

The dye accessibility of a fibre depends on— (1) the surface area through which the dye passes into the fibre,

(2) the speed at which the dye migrates in the fibre, (3) the internal volume v (c.c./g. dry fibre) in which the dye exists, and (4) the dissociation energy E of the dye–fibre complex. (1) and (2) can be calculated when E is known. If in the hot, very dilute direct dye solution, the sizes of the dye particles are very small and uniform, the following relation holds—

$$E = \frac{kT}{0{\cdot}4343} \log \frac{(m_0 - m)v}{mV}$$

(k = Boltzmann's constant; T = absolute temp.; m_0 = initial No. of dye particles in dyebath; m = No. of dye particles in the equilibrium dyebath; V = volume of dyebath). Dyeing experiments were carried out on viscose and cuprammonium rayons, Vinylon (a polyvinylformal fibre), and polyvinyl alcohol (P.V.A.) fibre at 80°C. for 3 hr. at a liquor ratio of 250 : 1 with Benzopurpurin 4 BKX (I), Chrysophenine NS (II), and Naphthamine Blue 12 B (III) (all purified).

Dye concn.— 0·001% (Wt.) (Benzopurpurin 0·003%)

NaCl concn.— 0·001% (Wt.) (Benzopurpurin 0·0003%)

The values for E (cal./particle $\times 10^{21}$) (see table) show that, under the same dyeing conditions, E of Vinylon is greater than that of cellulose fibres, but smaller than that of P.V.A. fibres.

Dye	Viscose Rayon	Cupram-monium	Cotton	Vinylon	P.V.A.
I	7·9	7·9	8·6	9·9	10·4
II	4·8	4·9	5·7	7·8	8·5
III	5·7	5·6	6·0	6·4	8·1

AUTHORS

Improvement in the Wet Fastness of Direct Dyeings, particularly by Aftertreatment with Cationic Agents

H. G. Büddicker-Löns

Z. ges. Textilind., **58**, 93–97 (5 Feb. 1956)

Methods for improving the wet fastness properties of direct dyeings and white discharges on direct-dyed grounds are detailed with special reference to cationic products such as Levogen WW, HW, and FW (FBy). Aftertreatment of Benzocuprol and Benzocupren (FBy) dyes with copper-containing cationic cpd., Benzocupren B and Benzocuprol WL (FBy), is described. B.K.

Ice for Uniform Package Dyeing

W. R. Jones

Text. Industr., **119**, (8), 98–99 (1955): *J. Textile Inst.*, **47**, A 34 (Jan. 1956)

In an American dyehouse the warm supply water, especially in summer, was responsible for uneven dyeings when using vat, direct, and developed dyes. The problem was solved by the addition of ice to the dye vessels. Some package dyeing procedures using ice are described. C.J.W.H.

High-temperature Vat Dyeing with Sodium Sulphoxylate instead of Sodium Dithionite

P. J. Wood

XXVII Congr. intern. Chim. ind., Bruxelles, **3**, 452–453 (1954): *Chem. Abs.*, **50**, 2175 (10 Feb. 1956)

Using one-third the weight of sulphoxylate compared with that of dithionite normally used in ordinary methods of dyeing yielded, by the high-temperature method, excellent levelness and penetration in package dyeing of yarn and hosiery. Use of sulphoxylate alone also permits better control of the rate of exhaustion of the dye. Many dyes liable to be over-reduced when dyed by conventional methods can be safely used by this process even at or near the boil. (cf. J.S.D.C., **70**, 591 (1954).) C.O.C.

Application of Conductivity Measurements in Dyeing with Vat Dyes

H. Borsten and F. L. J. van Lamoen

Tex, **14**, 1247–1252 (Sept. 1955): *J. Textile Inst.*, **47**, A 36 (Jan. 1956)

By controlling the alkalinity of the dyebath by means of conductivity measurements in combination with potential measurements, dyeings are obtained that can be satisfactorily reproduced (especially in the case of high-temperature dyes), and considerable savings in hydrosulphite can be effected. C.J.W.H.

Redox Potential and its Importance in Continuous Vat Dyeing

K. Neufang

Canadian Textile J., **73**, 51–52 (27 Jan. 1956)

Translation of paper published in *Melliand Textilber.*, **35**, 1012–1014 (Sept. 1954): J.S.D.C., **70**, 591 (Dec. 1954).

Continuous Dyeing with Esters of Leuco Vat Dyes

G. Strohbach

Textil Praxis, **10**, 1151–1157 (Nov. 1955)

Practical details for the application of Anthrasol (FH) vat dyes by a padding technique. B.K.

Warp-pile Fabrics

G. Capron

Teintex, **21**, 108–125 (Feb.), 205–217 (March 1956)

After a short historical introduction, the various types of fabric are defined and their dyeing and finishing described, with practical details of scouring, chlorination, and acidification. Dyeing on slow jigs with direct dyes and printing are discussed. Waterproofing, fireproofing, and crease-resist finishing are referred to in some detail. The second part is devoted to recommendations for the dyeing and finishing of mohair plushes and imitation astrakhan. S.R.C.

Influence of Dyeing on the Mean Diameter of Wool Fibres

M. Robinet and F. Monfort

Ann. Sci. Text. Belges, (4), 121–130 (1955): *J. Textile Inst.*, **47**, A 88 (Feb. 1956)

Variations in the weight, volume, density, and fibre diameter of acid-dyed wool have been investigated, using the projection microscope and air-flow methods (*J. Textile Inst.*, **45**, P 312–P 316 (1954)). The observations have shown that statistically significant differences do not exist between the fibre diameters of dyed and undyed material. C.J.W.H.

Dyeing of Metal-complex Dyes with Preservation of Wool

M. Robinet and N. J. Michailidis

Teintex, **21**, 187–195 (March 1956)

Wool dyed with metal-complex dyes was examined by the Krais–Viertel–Markt (K.M.V.) test and by load–extension measurements on single fibres and on yarns. A dyeing in 4% H_2SO_4 at 100 : 1 liquor ratio shows noticeable loss of tenacity after 1 hr. at the boil. After 4 hr. with 8% H_2SO_4 the loss is alarming. The damage also renders the wool more sensitive to subsequent and potentially damaging treatments. S.R.C.

Application of Weakly Acid-dyeing Metal-complex Dyes

R. Keppler

Textil Praxis, **11**, 270–274 (March 1956)

A study is made of the pH of the wool and dye liquor in the application of weakly acid-dyeing metal-complex dyes by the normal methods, and the results of these investigations have proved some guide to bulk application. Estimations of alkali solubility, acid solubility, cystein content, and solubility in urea–bisulphite soln. were considered to be necessary in order to ascertain wool damage. B.K.

Dyeing and Heat Treatment of Arnel (Triacetate)

F. Fortess, W. J. Myles, V. S. Salvin, and W. A. Shoeneberg

Amer. Dyestuff Rep., **45**, P 88–P 94, P 105 (13 Feb. 1956)

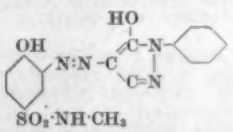
The properties of Arnel triacetate fibre are fully discussed with particular emphasis on dyeing. Resistance to saponification is good, and is of value in blends with cotton, which can be mercerised and then dyed with easily reducible vat dyes with negligible staining of the triacetate. Short liquors are desirable, and with disperse dyes good results are obtained at 4 : 1 liquor ratio with addition of 10% (on wt. of fabric) of emulsified tripropyl phosphate. The effect of heat aftertreatment and/or tripropyl phosphate in the dyebath in promoting dye penetration into the fibre is illustrated by photomicrographs, and methods of briefly applying high temperatures (up to 450°F.) are discussed. Dyeing of blends with cotton, viscose rayon, wool, nylon, and Terylene is surveyed and full recipes are provided. J.W.B.

Effect of Dyes on the Colloidal Properties of Aqueous Methylcellulose— I
S. Inokawa
J. Chem. Soc. Japan, Pure Chem. Sectn., **76**, 726–729 (1955): *Chem. Abs.*, **50**, 3755 (10 March 1956)
Almost all direct dyes, but not acid or basic dyes, gel the solution, probably because the micelles of the dye cross-link the molecules of methylcellulose. C.O.C.

Interactions between Direct Dyes and Hydroxylated Polymers
G. Centola, D. Borruso, and G. Prati
Gazz. chim. ital., **85**, 1468–1486 (1955); *Tinctoria*, **52**, 341–348 (Sept. 1955): *Chem. Abs.*, **50**, 3764 (10 March 1956)
Presence of methylcellulose or polyvinyl alcohol displaces the height and the position of the light-absorption maximum. Electrolytes amplify this phenomenon; in absence of methylcellulose the same electrolytes have an opposite effect. That the dyes form addition compounds with the polymers is confirmed if the reversible association of dye molecules is regarded as the cause of changes in the absorption spectra. The dye is distributed between the water and the polymer (regarded as two molecularly dispersed phases) in a proportion depending on the forces of attraction between polymer and dye and on the solubility of the dye in the aqueous phase. The smaller degree of crystallinity of films made from methylcellulose solutions containing dyes compared with that of films made from methylcellulose free from dye is confirmed by molecular association between the two solutes. Direct addition of dyes to aqueous methylcellulose increases the viscosity of the solution and in some cases flocculates the methylcellulose. The increase in viscosity so caused is negligible for very dilute solutions, but is of considerable magnitude with strong solutions. Probably it is caused by the dyes cross-linking the methylcellulose molecules when a certain concentration of dye is attained. This is confirmed by ultracentrifuge sedimentation measurements of solutions of methylcellulose and dyes with which, under certain definite conditions of concentration, two velocities are observed, one of which corresponds to sedimentation of simple macromolecules and the other to sedimentation of groups of macromolecules cross-linked to one another. C.O.C.

PATENTS

Dyeing Wool, Nylon, etc. with Metal(Cobalt)-complex Monoazo Dyes from Baths containing Phosphates
Ciba *BP* 745,410
Cobalt complexes of monoazo dyes containing 1 Co to 2 mol. of metallisable monoazo dye, free from SO_2H and COOH groups but containing a sulphamyl or sulphone group, give purer and stronger dyeings on animal fibres and nylon if the dyebath contains anions derived from $H_{n+2}P_nO_{3n+1}$ (n = integer). The pH of the dyebath is maintained at 4–9, preferably 6–8. Thus loose wool is treated at 50°C. for a short time in a bath containing a mixture of 70 parts of $NaH_2PO_4.H_2O$ and 30 parts of $Na_2HPO_4.12H_2O$ at a concn. of 2 g./litre, which gives a pH of 6·3. The metal-complex dye from 1 atom of Co and 2 mol. of—



is then added, and the bath is boiled for 45 min. to give a level brownish-yellow dyeing. E.S.

Decreasing the pH of the Bath during Dyeing
Albright & Wilson *BP* 746,474
Sodium monochloroacetate used in an acid or chrome dyebath hydrolyses, so that, using 5% on the wt. of the goods, there is the desired fall in pH during dyeing. Other α-monohalogenomonocarboxylic acids of 2–4 C also may be used. C.O.C.

Hair Dyeing
Gillette Industries *BP* 745,532
The hair is treated with 1 : 2 : 4-trihydroxybenzene during or before treatment with a primary aliphatic or arylaliphatic amine at pH 7–12 in absence of sulphites, hydrosulphites, and aliphatic thiols. C.O.C.

Dyeing Fabric on Beams (I p. 294)
Dyes and Dyeing in Ancient Mesopotamia (IV p. 295)
Comparison of the Stability of Some Intramolecular Hydrogen Bonds (IV p. 296)
Substantivity of Benzanilide Dyes (IV p. 298)
Water-soluble Sulphur Dyes (IV p. 298)
Influence of Ultraviolet Rays on the Tensile Strength of Cotton Fabrics (VI p. 303)
Colour and Finishing Problems in Dry Cleaning (VII p. 304)
Pressure Dyeing and Drying in a Closed System as compared with Dyeing and Drying in an Open System (X p. 307)
Transition Swelling in the Alkali Treatment of Cotton (X p. 307)
Simultaneous Hardening, Felting, and Cleaning or Dyeing of Wool Batts (X p. 309)
Classification of Dyes by their Dyeing Characteristics (XIV p. 312)

IX— PRINTING

Colloresin Printing Process
G. Bernardy and W. Küppers
Textil Praxis, **10**, 1137–1146 (Nov. 1955)
A detailed discussion of the Colloresin printing process for vat dyes, in which the material is first printed with a printing paste which does not contain any reducing agent, then padded in a solution of the reducing agent, and subsequently steamed. Particular attention is paid to thickeners and other components of the printing paste. B.K.

Monyl, Melamine, and Polyvinyl Alcohol in Screen Printing
R. Künzl
Textil Praxis, **11**, 179–181 (Feb. 1956)
Polyvinyl alcohol is finding wide use in place of gelatin in the preparation of screens by photoelectrical methods. It can be applied cold, in contrast to gelatin, and is readily removable from metallic and nylon screens. Melamine lacquers are finding increasing application, as they are fast to acids and alkalis, and are insol. in the most important solvents used in screen printing. Polyamide fibres are useful for making screens, as they are very strong and of good shear strength, do not swell, and possess good elasticity. As a result, very fine yarns can be used. Monyl (Züricher Beuteltuchfabrik A.G.) is a nylon material made of smooth single threads, specially suitable for making screens. B.K.

PATENTS

Metal-complex Azo Dyes as Printing Assistants in the Application of Thioindigoid Vat Dyes
American Cyanamid Co. *USP* 2,708,150
Azoic dyes metallised with a ferrous salt are used as printing assistants in aqueous printing pastes. By their use strong, smooth, bright prints are obtained. Thus Pigment Scarlet (*C.I.* 216) (0·25 mole) was dissolved in water (250 parts), the soln. stirred while slowly heating to 140–160°F., and then hydrated Na acetate (0·05 mole) added. $FeSO_4$ (0·025 mole) was added, and the mixture stirred at the boil for 30 min. The metallised dye was salted out, filtered, washed with dil. brine, and air-dried. The product used as an assistant in a printing paste containing the dye, bis-2:1-naphthioindigo, resulted in prints being obtained which were 20% stronger than those obtained in its absence. C.O.C.

Printing on Vinyl Plastics
IC *USP* 2,708,188
Compositions containing a pigment, a resin modified with an alkyl methacrylate as vehicle, and a vinyl chloride–acetate copolymer as the binder yield prints of good adhesion, flexibility, and freedom from tackiness on drying on vinyl plastics. Preferably they are used as water-in-oil emulsions, the binder being in the oil phase. C.O.C.

Electrostatic Printing
O.-B. Rasmussen *BP* 745,500
A dye is atomised into a mist of electrically charged particles, which is caused to migrate towards the carrier by the electrostatic field between an electrode charging the particles and an electrode charging the carrier. Local ionisation is produced in the carrier to form conductive and insulating parts on it in accordance with the desired pattern. C.O.C.

Colour Photography— Styryl Dyes as Dischargeable Yellow Filters
ICI *USP* 2,709,136
A non-diffusible styryl dye is used in at least one filter layer as a dischargeable yellow filter dye, the exposed and developed material being treated with an alkylamine, aralkylamine, hydrazine, alkylhydrazine, aralkylhydrazine, arylhydrazine, alkylenediamine, or hydroxylamine. The styryl dye may be obtained by condensing an anil of a substituted benzaldehyde with a reactive methylene compound. C.O.C.

Colour Photography
DuP *BP* 746,804
A simplified way of making differentially sensitised polyvinylacetal emulsions for multilayer colour films and papers so that the silver halide grains exhibit similar size distribution in each layer. C.O.C.

Colour Photography
Agfa A.-G. für Photofabrikation *BP* 746,567
As bleaching and fixing agent there is used a mixture of a silver halide fixing agent and a complex salt of an alkali metal and tervalent iron with an acid of formula—

$$\text{HOOC}\cdot R^2\cdot N(\cdot R^3)\cdot R^4\cdot\text{COOH}$$

$$\text{HOOC}\cdot R^1\cdot X\cdot R^2\cdot\text{COOH} \text{ or } \qquad R^5$$

$$\text{HOOC}\cdot R^3\cdot N(\cdot R^5)\cdot R^4\cdot\text{COOH}$$

(X = C, O, S, or NR^6; R^1–R^6 = subst. or unsubst. hydrocarbon radicals, or R^6 = H, polyethylene oxide, and/or a polyethylene oxide derivative). C.O.C.

Non-diffusing Polymeric Reducing Agents for Colour Photography
Eastman Kodak Co. *USP* 2,710,801

Coloured Photographic Reproductions by Image-wise Imbibition
Kodak *BP* 746,686

Photographic Colour-separation Records for preparing Printing Plates
O. E. G. Thorslund *BP* 746,574

Colour Digest. Colour Developers and Couplers— Review of Patents (IV p. 296)
Warp-pile Fabrics (VIII p. 305)
Wall Paper (XI p. 311)

X— SIZING AND FINISHING

Pressure Dyeing and Drying in a Closed System as compared with Dyeing and Drying in an Open System
F. Thies
Amer. Dyestuff Rep., **45**, P 95–P 98 (13 Feb. 1956)
Drying of cross-wound packages is discussed: methods employing tray dryers and hot-air circulation dryers of the open and closed types are surveyed, a new high-pressure closed-circuit dryer in detail. This circulates air at 85 lb. per sq.in. and incorporates a condenser system for preventing excessive moisture circulation, together with a control to prevent excessive drying and a flow-reversal system to ensure even drying. Static pressure dyeing is more briefly discussed. J.W.B.

Effect of Colloidal Silica Treatments on Sizing and Finishing Characteristics of Cotton Yarns
H. M. Brown, J. H. Langton, and E. A. Murray
Text. Research J., **26**, 40–43 (Jan. 1956)
The presence of 0·12% of colloidal silica on medium and fine carded yarns does not affect the operations of mercerising, bleaching, and dyeing. Yarn strength differences between treated and untreated samples were of the same order after as before each of these processes. Silica-treated slashed medium yarns, but not coarse yarns, showed improved abrasion resistance. Take-up and loss of size were found to be greater for fine untreated yarns than for comparable treated yarns; no similar difference was found for medium and coarse yarns. Determination of ash content is not a sufficiently sensitive test to assess the resistance of silica treatment to chemical processing. J.C.F.

Modern Resin Finishing of Textiles
G. Landells
J.S.D.C., **72**, 137–146 (April 1956)
The lecture surveys the development of the application of synthetic resins to textiles and the interrelation of practice and research. Resin application developed first on two main lines, viz. the use of emulsions of preformed vinyl-type polymers and the urea–formaldehyde crease-resist process. By using different preformed vinyl polymers a wide variety of finishes may be obtained, whereas the urea–formaldehyde finish, which is a key finish both practically and theoretically, is itself the basis of many finishes and has been the pattern for many other types of amino-formaldehyde resin finish. The importance of the location of the resin in or on the fibre is demonstrated in numerous systems. The use of resins for modifying handle, for imparting crease-resistance or dimensional stability, or for fixing embossed effects is now standard practice, but synthetic resins are finding increasing use in new outlets. Thus, phosphorus compounds are now being incorporated with the amino-formaldehyde type of resins to produce anti-flame finishes. Research is proceeding in the field of isocyanate chemistry, especially on polymer formation in and on textile fibres, and some progress is being made in practical application. A new class of polymeric substances— the silicones— has opened up a completely new field in the progress of water-repellency. It is shown that resin application to textiles is now a specialised study and that many more new developments may be expected. AUTHOR

Transition Swelling in the Alkali Treatment of Cotton
K. Messik and K. Szabó
Faserforsch. und Textiltech., **7**, 89–93 (Feb. 1956)
Experiments on swelling of cotton treated with NaOH soln. of varying concn., followed by rinsing with water or alcohol, show the influence of transition swelling on the degree of swelling of cellulose. Absorption of dye increases with NaOH concn. up to an alkali concn. of about 20%, and may then decrease or increase slightly or remain constant. The amount of amorphous material increases with NaOH concn., and this is partly responsible for the influence on dyeability. W.R.M.

Catalysis in Blended Chemical Finishes
D. D. Gagliardi
Amer. Dyestuff Rep., **45**, P 99–P 104 (13 Feb. 1956)
Finishing treatments requiring catalysts are increasing in number, and simultaneously applied reagents may require incompatible catalysts, e.g. an alkali-catalysed ketone resin applied with an acid-catalysed melamine resin. Moreover, ideal catalysts may have undesirable colour or toxicity. The theory and the mechanism of catalysis in textile finishing are surveyed, the finishes being — thermosetting formaldehyde-type resins, cationic softeners, dye-fixing agents, silicone emulsions, non-durable water-repellents, and resin-base water-repellents. Application of e.g. a water-repellent crease-resist finish may require six reagents, and it is difficult to trace the source of any subsequent fault. Afterwashing is of particular importance, to remove catalyst by-products. Experiments are described in which different resins are applied each with a number of catalysts, and the tensile changes in unwashed fabrics due to curing and to subsequent scorching are measured. J.W.B.

Variables of Cure in the Resin Finishing of Cotton
S. H. Foster
Text. Research J., **26**, 149–155 (Feb. 1956)
The effect of temperature of cure, time of cure, and amount of catalyst on the crease recovery, tensile strength, and tear strength of cotton fabric treated with varying amounts of (i) cyclic dimethylolethyleneurea and (ii) methylated trimethyolmelamine has been studied. Curing temperature exerts the most important effect. A higher curing temperature is required for (ii) than for (i). Resin (i) gives better crease recovery than (ii), but for samples cured at the same temperature (ii) shows less tensile loss. Increasing the time of cure from 1·5 to 6 min. has no effect on the physical properties studied for either resin. Variation

of the amount of catalyst has a slight effect, particularly at low concentrations of resin solids, but the effect falls off when the amount of catalyst is $> 10\%$ of the resin solids. Both resins, when properly cured, showed good resistance of physical properties to washing. J.C.F.

Characteristics of "Wash-and-wear" Cottons
A. L. Lippert
Text. Research J., **26**, 128–135 (Feb. 1956)

The properties desirable in minimum-care cotton fabrics are discussed. Fabric construction to promote high crease recovery and good draping qualities is important. Relaxation shrinkage of the fabric imparts dimensional stability and improves crease recovery and washing performance. Resin treatments, primarily applied to improve dimensional stability, also influence chlorine retention, ironing temperature, and water-repellency. J.C.F.

Mechanical Properties of Cellulose Fabrics treated with Mono- and Bis-hydroxymethylureas (Mono- and Di-methylolureas)
R. Steele and L. E. Giddings
Text. Research J., **26**, 116–123 (Feb. 1956)

The load–elongation properties, elastic stiffness, and stress relaxation of cotton and rayon fabrics treated with dimethylolurea (I) and monomethylolurea (II) have been examined. The elastic stiffness of the fabrics is not altered by resin treatment, and the principal effect is a reduction in the rate and the extent of flow and slippage occurring during deformation, leading to an increase in tensile recovery and therefore in crease resistance. I, which is a cross-linking agent, is much more effective for cotton than II, a non-cross-linking agent. On rayon, I and II are about equally effective. This difference in behaviour is related to the difference in orientation of the two fibres. J.C.F.

Crease-resist Finishing of Linen Fabrics
M. Orzel, Z. Machnowska, and L. Wierzbowska
Prace Inst. Wlok., **4**, (17), 54–58 (1955): *J. Textile Inst.*, **47**, A 39 (Jan. 1956)

Linen fabrics, impregnated with urea–formaldehyde resin, showed improved crease-resistance, increased dimensional stability (decreased shrinkage after washing), reduced swelling in water, and, therefore, increased drying rate. The tensile strength of the fabric decreases proportionally to the improved creasing properties, but gradually increases again with the removal of the finish during washing. The urea–formaldehyde resins are not fast to washing on boiling, but under milder washing conditions (i.e. at about 60°C.) they are very stable, and about 25% of the resin still remains on the fabric after 15 launderings. C.J.W.H.

Rotproofing of Viscose Rayon. II—Treatment of Viscose Rayon with Iron and Chromium Salts
J. W. Bell and M. M. Ramsey
J.S.D.C., **72**, 168–171 (April 1956)

Although "Mineral Khaki" treatments are recommended for proofing cotton against micro-organisms, certain types of chromium and iron pigmentation are shown to be relatively ineffective when applied to viscose rayon, at least when fairly drastic test methods are employed. This is in agreement with results previously obtained with cotton. The proof is slightly improved by using potassium chromate as the developing agent, but the sexavalent chromium produced is not fast to leaching. Little improvement is effected by incorporating copper. The viscose rayon takes up considerably less pigment than does cotton under similar conditions, but even where the amounts are almost the same the rayon is still much less resistant to micro-organisms. This difference may be partly due to the lower inherent resistance of viscose rayon. When chromate development is used the rayon appears to be less damaged (as measured by cuprammonium fluidity) than cotton similarly treated. AUTHORS

Separation of Formaldehyde during Ironing of Impregnated Fabrics
W. Vauck
Textil- und Faserstoff-tech., **5**, (9), 529–531 (1955): *J. Textile Inst.*, **47**, A 53 (Jan. 1956)

On damp-ironing fabrics impregnated with carbamide resins, separation of the formaldehyde takes place, which can be determined with 0·025 N. thiosulphate solution with an accuracy of $\pm 10\,\mu g$. The method is described. Experiments have shown that all fabrics condensed with carbamide resins at 100–180°C. for various lengths of time split off formaldehyde during damp-ironing, the amount decreasing with increasing temperature and duration of condensation and storage time. The fabrics should be rinsed after impregnation and stored at room temperature and humidity in a well ventilated place, where the after-condensation proceeds with further separation of formaldehyde. Unrinsed fabrics should be stored for at least four weeks. Steam-decatising is equivalent to the rinsing process, reducing the storage time necessary for the treated fabrics, prior to making up, to two weeks. C.J.W.H.

Effect of Chlorine and Subsequent Treatment with Sodium Bisulphite on Physical Properties of Cotton Fabrics treated with Various Resins
J. H. Howard
Text. Research J., **26**, 124–128 (Feb. 1956)

Fabrics treated with thermosetting resins and subsequently laundered in the presence of small amounts of chlorine are liable to retain the latter. The present study relates to the effect of chlorine treatment and subsequent dechlorination with $NaHSO_3$ on the physical properties of cotton fabric treated with the following types of resin—urea–formaldehyde (I), melamine–formaldehyde (II), and iminazolidone–formaldehyde (III). Retained chlorine causes a loss in crease resistance of fabric treated with all three resins and a reduction in weft tearing strength of I- and II-treated fabrics. $NaHSO_3$ may be used to remove retained chlorine, but losses in crease resistance persist, being lowest for III-treated fabric. Chlorine–bisulphite treatment causes a reduction in weft tearing strength of I- and III-treated fabrics, and an increase in the weft tearing strength of II-treated fabric. Laundering in alkaline soap solution causes a loss in crease resistance and weft tearing strength of fabric treated with all three resins. J.C.F.

Changes produced by the Heating of Cotton Cellulose
R. L. W. Farquhar, D. Pesant, and B. A. McLaren
Canadian Textile J., **73**, 51–56 (10 Feb. 1956)

Raw cotton subjected to heat treatment at temp. of 50–220°C. for times of 4–24 hr. in air and in nitrogen has been analysed by various methods. The conclusions reached from a study of cuticle degradation, weight loss, fluidity, carboxyl and hydroxyl contents, and enzymatic hydrolysis are—(1) heat alone accounts for only a part of the degradation of cellulose; (2) oxidative action begins at lower temperatures, and accelerated thermal decomposition at higher temperatures; (3) cuticle degradation appears to be a function of combined thermal and oxidative damage and not the result of heat alone. A.H.

Compounded Flame-retardant for Cotton Fabrics
J. D. Reid, J. G. Frick, and R. L. Arceneaux
Text. Research J., **26**, 137–140 (Feb. 1956)

A combination of two existing flame-retardants, the one a copolymer of tetrakishydroxymethylphosphonium chloride (THPC) and methylolmelamine (cf. J.S.D.C., **70**, 474 (1954); **71**, 564 (1955)), and the other a bromoform–triallyl phosphate polymer (cf. J.S.D.C., **71**, 427 (1955)), gives a durable flame-retardant finish for cotton superior to that given by either alone. The treatment is applied from a single bath, and the finishing operations required are simple. J.C.F.

Bromine-containing Phosphonitrilates as Flame-retardants for Cotton
C. Hamalainen and J. D. Guthrie
Text. Research J., **26**, 141–144 (Feb. 1956)

The following bromine-containing derivatives of phosphonitrilic chloride have been investigated as possible flame-retardants for cotton: brominated allyl phosphonitrilate (I), 2:3-dibromopropyl phosphonitrilate (II), and the bromoform adduct polymer of allyl phosphonitrilate (III). All were found to give effective flame-retardant finishes durable to detergent laundering. Comparative tests indicate that III is most suitable for further trials. J.C.F.

Cotton made Flame-resistant with Bromine-containing Phosphonitrilates in Combination with Tetrakishydroxymethylphosphonium Chloride (THPC) Resins
C. Hamalainen, W. A. Reeves, and J. D. Guthrie
Text. Research J., **26**, 145–149 (Feb. 1956)

An efficient permanent flame-retardant finish for cotton fabrics is obtained by use of a bromoform adduct polymer

of allyl phosphonitrilate in combination with THPC resin (see two preceding abstracts). J.C.F.

Theory of Flame-retardant Finishes
I. M. Gottlieb
Text. Research J., **26**, 156–167 (Feb. 1956)

The author summarises recent findings on the mode of action of flame-retardant finishes on cotton, e.g. the rapid evolution of volatile condensable products at a comparatively low temperature; the necessity for the presence in the flame-retardant of a group, such as the orthophosphate group, which will promote catalytic dehydration of the cellulose substrate to carbon and water; the possibility of reducing the inflammability of the volatile products by incorporating a halogen, particularly bromine, in the flame-retardant molecule. These considerations lead to a discussion of the types of compound which may be profitably examined for flame-retardant properties. The inadequacy of present methods of testing flame-resistance is illustrated. Laboratory evaluation should take into account— (*a*) the characteristics of the base fabric, (*b*) the nature of the source of ignition, and (*c*) the specific demands which the fabric would satisfy in use. J.C.F.

Recent Progress in the Wool Industry
E. G. H. Carter
Teintex, **21**, 195–202 (March 1956)

The sheep-marking lanolin-based emulsion fluids, anti-static treatments, damage in worsted yarns, the Maifoss and Manofast processes, and shrinkproofing by Epilox and Si-Ro-Fix are reviewed. S.R.C.

Chemical Bases of the Felting and Milling of Protein Fibres
E. Elöd and W. Meyer
Textil Praxis, **11**, 163–170 (Feb. 1956)

After discussing some of the published work and theories regarding the felting of protein fibres on milling, the paper deals with the factors affecting the curling and the contraction of fibres, particularly rabbit fur, and details the influence of carrotting solutions. Experiments are described which study the effect of hydrogen peroxide, nitric acid, and formamide in carrotting rabbit fur. B.K.

Modern Methods of Electrostatic Flocking of Textiles, Paper, etc.
A. Franken
Tintorería Industrial, **2**, 267–285 (Sept. 1955)

The outlets, methods, machinery, and adhesives are described. S.R.C.

PATENTS

Crimping Yarn
Billion & Cie. *BP* 746,410

The yarn is wound at least once round a support, drawn by one end while so wound, and heat-set while it is being drawn. C.O.C.

Permanently Crimped Polyamide or other fully Synthetic Yarn
Heberlein & Co. *BP* 746,992

Temporary high twist is given to initially smooth yarn having little or no twist and then it is set by wet and/or hot treatment. The rate of withdrawal of the yarn from the twister of the false-twisting device is at least 0·5% greater than the rate of feed of yarn to the twister. C.O.C.

Siliceous Coatings on Textiles
Monsanto *BP* 746,483

The material is treated with an ethyl silicate and then with a gaseous or vaporous hydrolysis catalyst. Thus 30-denier nylon hose were treated in an aqueous emulsion produced with Silester O (Monsanto) and Lubrol W (ICI) and then with ammonia vapour. This greatly increased their resistance to snagging. C.O.C.

Crinoline
Johnson & Johnson *USP* 2,712,312

Crinoline, commonly used as protective strip for adhesive bandages, etc., is produced by impregnating woven gauze with an aqueous dispersion of a modified starch together with an aluminium salt (<0·25% Al on wt. of starch). It has less tendency to fusion of fabrics than ordinary crinoline during heat-sterilising. C.O.C.

Reducing the Porosity of a Fabric by "Steam Blasting"
Goodyear Tire & Rubber Co. *USP* 2,712,170

The wet fabric while confined or restrained in its cross-sectional dimensions is subjected to the instantaneous application of a relatively high temperature. This causes "steam blasting" and quick volatilisation of the liquid in the fibres, so that the internal forces set up in the yarns cause them to spread and flatten. C.O.C.

Resin Finishing of Regenerated Cellulose Fabrics
Kendall Co. *USP* 2,709,141

If a regenerated cellulose fabric is impregnated with an aqueous solution of a resin precondensate in presence of a polymerisation catalyst and is kept in the wet state for <10 min. before drying and baking, then the amount of precondensate which can be applied may be increased to much more than that which, if the fabric were dried in <10 min. after impregnation, would cause the final fabric to be stiff and brittle. C.O.C.

Coating or Impregnating with Synthetic Resins
FBy *BP* 745,960

In coating or impregnating with the high-mol.wt. polymers obtained by reaction of a polyester or a polyester amide with a *di*isocyanate, addition to the composition of an *iso*cyanate-modified ester containing free NCO groups, as a cross-linking agent, renders the cured coating non-thermoplastic and insoluble in organic solvents. C.O.C.

Quick-setting Proteinaceous Coatings
Eastman Kodak Co. *USP* 2,710,815

When aqueous solutions of proteins, particularly gelatin, are applied to paper or cloth, they may be immediately set if the coating soln. contains a soluble aluminium salt, and a gas containing a small proportion of ammonia is applied to the coated material. C.O.C.

Aqueous Halogenoalkyl Phosphate–Amino Resin Flame-resisting Compositions
U.S. Secretary of Agriculture *USP* 2,711,998

Textiles are impregnated (10–25% of solids) with an aqueous solution containing (1) an ammonium salt of a polychloro- or polybromo-propanol acid ester of orthophosphoric acid, (2) a water-soluble amino-formaldehyde resin precondensate, and (3) enough NH_4OH to give pH > 7, dried at 60–65°C., and baked for 3–8 min. at 140–150°C. Thus bis-2:3-dibromopropyl phosphate, 1 part of a melamine–formaldehyde (1 : 3 mol.) precondensate, and NH_4OH to bring to pH > 7 were dissolved in 4 parts of water. An 8·2-oz. cotton cloth was impregnated with this solution to a 24% solids pick-up, dried at 60–65°C., and baked at 150°C. for 5 min. It was then flame-resistant. C.O.C.

Imparting Wool-like Finish to Cellulosic Fabrics
Joseph Bancroft & Sons Co. *USP* 2,709,638

The fabric is treated so as to form a nitrogenous cellulose–acid complex, e.g. by impregnation with an aqueous solution of orthophosphoric acid and urea and heating, after which it is treated with NaOH so that the H atoms in the complex are replaced by Na. Thus cotton cloth was impregnated with a solution prepared from 75% orthophosphoric acid (60 lb.), dicyandiamide (40), urea (120), 28% ammonia (60), 37% H·CHO (80), and water (to bring to 100 gal.(U.S.A.)), dried, baked at 320°F. for 6 min., washed, and dried. It was then treated on a mercerising machine with 60°Tw. NaOH, washed with warm water while under tension on the machine, treated with aq. $NaHCO_3$ in the wash boxes, rinsed, dried, and finally thoroughly impregnated with 2% aq. Ahcovel G, squeezed, and dried. The finished fabric had excellent strength, very good recovery from creasing, and a flexible, full, soft, wool-like handle. C.O.C.

Simultaneous Hardening, Felting, and Cleaning or Dyeing of Wool Batts
M. T. Hoffmann *USP* 2,712,171

A method of simultaneously hardening, felting, and cleaning or dyeing wool batts in a multiroller machine. C.O.C.

Treating Non-woven Cellulosic Fabrics with Weakly Acid Aminotriazine Resin Colloids
American Cyanamid Co. *USP* 2,708,644

The strength of non-woven cellulosic fabrics, most of whose fibres are oriented in the same direction, is increased by treatment with a weakly acid, cationic aminotriazine–aldehyde resin colloid, e.g. an acetic-acid colloid of methylated trimethylolmelamine. C.O.C.

Weftless Fabrics
BrC *BP* 745,644

An open-width warp is treated with a long-chain condensation polymer whose chains contain more ester than amide linkages, after which the polymer is converted into a rubbery polymer by cross-linking. Thus an acetate-rayon warp was impregnated (so as to deposit 25% of solids) with a composition made up of Vulcaprene AC (ICI) (14 parts), methyl ethyl ketone (60), methyl *iso*butyl ketone (26), Vulcafor VHM (ICI) (0·7), and Vulcafor VDC (ICI) (0·1) at 35°c., dried, and baked at 140°c. for 10 min. C.O.C.

Protecting Cellulosic Fibrous Materials of Low Swelling Capacity from Attack by Micro-organisms
Ciba *BP* 745,638

The materials are impregnated with an aminoplast precondensate (3–10% aminoplast on wt. of goods), curing of the resin being then carried out in presence of water. Thus cotton yarn is impregnated with 170% on its wt. of a solution of 1 mol. melamine–3 mol. H·CHO precondensate (60 g.), H·COOH (24), non-ionic wetting agent (1), and water (to 1 litre), stored overnight in a closed vessel, rinsed first with dil. ammonia and then with water, and dried. Its mechanical, swelling, and dyeing properties remain unchanged, but it is now resistant to bacterial attack. C.O.C.

Reducing the Felting Power of Wool
Midland Silicones *BP* 746,307

A composition containing 25–95% by wt. of a benzene-soluble diorganopolysiloxane of viscosity $> 10^8$ centistokes at 25°c. and 75–5% of an organosiloxane of average formula $R_{2n}SiH_{2m}O_{4-(m+n)}$ (R = Alk, alkenyl, or phenyl; $n = 0{\cdot}5$–$0{\cdot}95$; $m = 0{\cdot}05$–$0{\cdot}5$; $n + m = 0{\cdot}825$–$1{\cdot}05$) is used to prevent wool felting during washing. It alters neither the appearance nor the handle of the treated material. C.O.C.

Coating Cloth or Paper with a Thermoplastic Material of High Melting Point
P. J. Massey *USP* 2,712,508

The difficulty of handling films of a thermoplastic material, e.g. polyethylene, which flows at $< 212°F$. and is extremely viscous at high temperatures, is overcome by feeding the thermoplastic into and across the entire length of a nip made by two rollers one of which rotates much more quickly than the other. The rollers are heated to avoid solidification of the plastic. The cloth or paper is caused by the faster moving roller to pass through and into contact with the plastic. C.O.C.

Coating Plastics and Plastics-coated Fabrics with Metal
ICI *BP* 745,819

When vacuum-coating metal on to flexible plasticised polymeric material, better adhesion is obtained if the plastic is first coated with a non-metallic material which is impermeable to the plasticiser or other component in the plastic which may be volatile under vacuum. Thus a base fabric was coated with cellulose nitrate plasticised with 40% on its wt. of castor oil. A further coating was given with a 20% soln. of nylon in 8 : 2 alcohol–water at 60°c. so as to deposit 0·3 oz. (dry wt.) of nylon per square yard. A film of aluminium was then deposited on top of the nylon by vacuum-coating. There was much better adhesion of the aluminium than in absence of the nylon layer. C.O.C.

Colloidally Dispersed Oxides for the Textile Industry (III p. 294)
Novel Method of Cyanoethylation. IV—Substitution Effects (IV p. 296)
Warp-pile Fabrics (VIII p. 305)
Dyeing and Heat Treatment of Arnel (Triacetate) (VIII p. 305)
Physicochemical Investigations of Cellulose and Related Carbohydrates (XI this page)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Brightness Reversion of Bleached Pulps. II—Alkali Extraction of Yellowed Material
H. W. Giertz and J. McPherson
Svensk Papperstidning, **59**, 93–97 (15 Feb. 1956)

Hypochlorite-bleached sulphite pulp was aged by heating at 110°c. for 40 hr., and after yellowing it was extracted with LiOH soln. The colour of both the α- and the β-cellulose obtained was white, whereas the γ-cellulose was orange-coloured. On further fractionation of the solution, a white, high-mol.wt. fraction was isolated from the orange and brown substances, which were found to be highly acidic and to have a high carbonyl content. If pulp is reduced with sodium borohydride soln. prior to ageing, the degree of yellowing appears to be decreased. It is assumed that the yellowing is caused by degraded hemicellulose products and that the carbonyl groups formed during bleaching are an important factor in the yellowing of pulp. K.W.

Relation between Hemicellulose Content of Pulp and its Paper Strength
N. Migita, T. Kanda, S. Sato, and Y. Kojima
J. Soc. Textile Cellulose Ind. Japan, **12**, 14–18 (Jan. 1956)

Ultraviolet Absorption of Cellulose
J. Schurz
Svensk Papperstidning, **59**, 98–103 (15 Feb. 1956)

Ultraviolet-absorption measurements of model cellulose substances did not provide a solution of the problem of the spectral characteristics of cellulose. A group of oxidised celluloses show, however, absorption maxima at 3770–3840 $mm.^{-1}$, and has been termed oxycellulose I (OI). Experiments have shown that OI is formed on oxidation with H_2O_2 and on ultraviolet degradation of native and regenerated cellulose. Depending on the chain length, OI is insoluble, alkali-soluble, or water-soluble. It is the main constituent of the so-called residual hemicellulose ("Resthemi", i.e. the extract obtained with 10% NaOH from pulp pre-extracted with 18% NaOH) and therefore mainly responsible for the damage to fibres caused by hemicellulose. In the yellowing of pulp, OI is formed initially under the action of light and oxygen, and is further degraded to deeply coloured, low-mol.wt. products. K.W.

Cupriethylenediamine as a Solvent for Cellulose
K. Wilson
Svensk Papperstidning, **59**, 88–92 (15 Feb. 1956)

Cupriethylenediamine (CED), containing 1 mol. Cu per litre, is prepared by saturating ethylenediamine soln. with $Cu(OH)_2$, giving an en : Cu ratio of 2·0 and high solvent power for all types of cellulose. If en : Cu > 2·0, the solvent power decreases rapidly. The relative viscosity of a cellulose–CED solution increases with increasing en : Cu ratio and decreases with increasing Cu content. The composition of the soln. can, however, be easily adjusted so that the variation in viscosity $> \pm 1\%$. K.W.

Heterogeneous Hydrolysis of Cotton and Sulphite Cellulose
J. Linderot
Svensk Papperstidning, **59**, 37–43 (31 Jan. 1956)

Cellulose hydrolysed under mild conditions shows a marked reduction in mol. wt. during an initial rapid reaction, which is followed by a slower reaction at almost constant mol. wt., the limit D.P. Differing views on the degradation mechanism are discussed. In the present investigation, the influence of cellulose fine structure on the degradation reaction and the limit D.P. value, and the change of crystallinity during hydrolysis, have been studied. Purified cotton and sulphite cellulose and regenerated cellulose prepared from these pulps were hydrolysed in N-HCl at 60°c. for periods up to 96 hr. From moisture sorption, X-ray, and mol-wt. measurements, it is concluded that the D.P. limit value of cellulose depends largely on the supermolecular structure of the material expressed as the ratio of crystalline to amorphous substance. Cellulose regenerated from cupreous solutions and dried from benzene contains amorphous regions that are not available for moisture sorption. Hydrolysis results in real crystallisation amounting to a max. of 29% for cotton cellulose and 15% for regenerated cotton and sulphite cellulose. 22 references. K.W.

Physicochemical Investigations of Cellulose and Related Carbohydrates
B. G. Rånby, E. H. Immergut, and H. F. Mark
Chem. Ber., **89**, 526–534 (Feb. 1956)

A review of the differences between the cellulose of cotton and of wood in X-ray diffraction, in mercerisation, and in degradation by heterogeneous and homogeneous hydrolysis, with 27 references. H.E.N.

Optimum Conditions for the Preparation of Carboxyethylcellulose
A. G. Yashunskaya, Z. A. Rogovin, and A. A. Berlin
J. Appl. Chem. U.S.S.R., **29**, 105–110 (Jan. 1956)

PATENTS

Improving the Wet Strength of Paper
Portals *BP* 744,383
A neutral emulsion of ammonium caseinate, methylhydroxycellulose, and tri- or hexa-methylolmelamine is applied to the paper web in tub sizing, with or without addition of gelatin, at the beater, or on the paper machine. Aluminium sulphate may be added to precipitate the size on the fibres. K.W.

Improving the Wet Strength of Paper
FBy *BP* 745,499
The wet tensile strength of paper is improved by adding to pulp or finished paper 1–3% of a water-soluble, high-mol.wt. basic condensate containing epoxy groups, obtained by reaction of epichlorohydrin (I) or dichlorohydrin (II) with a di- or poly-amine (< 16 C) (including amines prepared by reaction of I or II with excess of ammonia or monoamines) under conditions that allow further cross-linking of the products. K.W.

Wall Paper
Société Française des Papiers Peints *BP* 745,541
A cloud of metallic or non-metallic powder is blown along a printed paper strip passing through a distributing box adjoining a printing machine, so that the powder adheres to relief ink-printed patterns, excess powder being removed from uncoated areas by a suction brush as the paper leaves a dryer following the distributing box. K.W.

Carbon Paper
F. G. Francis *BP* 744,909
Carbon paper is coated on one or both sides with a 0·0005–0·001 in. thick polythene film, e.g. by extrusion coating. The toughness and the resilience of the film may be modified by the addition of poly*iso*butylene. White or coloured pigments and/or fillers to impart slight roughness may be incorporated in the film or coated on the extruded film. The layer of polythene prevents the tendency of paper to become dished during use. K.W.

Black Interleaving Paper for Packing Light-sensitive Materials
Eastman Kodak Co. *USP* 2,710,800
Paper made from pulp dyed with Dupont Fiber Black VF or Diamine Black Cap Extra Conc. (salt-free) (both are *C.I.* 581) shaded with Pontamine Yellow SXPE (DuP) (*C.I.* 622), or with Direct Deep Black EA Extra CF (*C.I.* 581) without a shading dye, is very suitable for wrapping light-sensitive materials. C.O.C.

Heat-sensitive Thermographic Duplicating Paper
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Reaction of Benzoquinonemonoimine with Lignin. II— Isolation and Identification of the Colouring Matters formed (IV p. 298)
α-Benzyl-*β*-methylumbelliferone— Fluorescent Brightening Agent for Paper (IV p. 302)
Lateral Order Distribution in Cellulose Fibres (VI p. 303)
Modern Methods of Electrostatic Flocking of Textiles, Paper, etc. (X p. 309)
Quick-setting Proteinaceous Coatings (X p. 309)
Coating Cloth or Paper with a Thermoplastic Material of High Melting Point (X p. 310)

XIII— RUBBER; RESINS; PLASTICS

PATENT

***p-tert.*-Butylphenol as Stabiliser for Pigmented Cellulose Acetate-Butyrate Moulding Compositions**
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Amine-coated Calcium Carbonate Pigments (IV p. 302)
Coating Plastics and Plastics-coated Fabrics with Metal (X p. 310)

XIV— ANALYSIS; TESTING; APPARATUS

Test for Formaldehyde
E. Mácciotta
Boll. lab. chim. provinciali (Bologna, Italy), **6**, (2), 49–50 (1955):
Chem. Abs., **50**, 2368 (25 Feb. 1956)
3:5:6-Trihydroxy-2-iodo-1-methylindole turns blue with HCHO in HCl. Other aldehydes give other colours. The reagent is prepared by warming adrenaline (1 g.) in methanol (50 ml.) to 40°c. and adding glacial acetic acid drop by drop until a clear solution is obtained. HIO_3 (1 g.) in water (5 ml.) is added at 40°c., when the solution turns blood red and forms needle crystals. These are filtered off, and washed with methanol and then with water. They are suspended in cold water (50 ml.), and $NaHSO_3$ (2 g.) dissolved in a minimum of water is added to bleach the solution. The crystals are filtered off, washed with ice-cold water, and dissolved in methanol. To this light yellow soln. 3:5-dinitrobenzoic acid (1 g.) dissolved in methanol is added. On dilution with water orange crystals form. These are recrystallised from benzene or chloroform. The sample to be tested is distilled. To the distillate (5 ml.) add 1 ml. of a 0·1% acetic-acid solution of the above crystals and 1 ml. of conc. HCl and heat. An intense blue develops if 100 *μg.* of H·CHO is present. C.O.C.

Determining Very Small Quantities of *p*-Phenylenediamine and its *NN*-Dialkyl Derivatives
W. Massmann and G. Menge
Z. anal. Chem., **148**, 100–105 (1955):
Chem. Abs., **50**, 3161 (10 March 1956)
A photometric method which, under certain conditions, ensures identification of $1{\cdot}0\text{–}2{\cdot}0 \times 10^{-9}$ to $2{\cdot}0 \times 10^{-7}$ mol. of *p*-phenylenediamine and its *NN*-dialkyl derivatives. C.O.C.

Molecular Chromatography. IV— Resolution of Mixtures of Nitroanilines
K. A. Ogloblin and G. V. Markina
J. Gen. Chem. U.S.S.R., **26**, 95–101 (Jan. 1956)
A mixture of 2:4:6-trinitroaniline with all the mono- and di-nitroanilines and other mixtures of selected groups of these components are satisfactorily resolved by chromatography in benzene soln. on slightly moist alumina (2–3% of water). A symbatic relationship is found between the dipole moment of the nitroaniline and its tendency to be adsorbed by alumina. A.E.S.

Polarography of Azo Dyes
J. de O. Cabral and H. A. Turner
J.S.D.C., **72**, 158–167 (April 1956)
Twenty-one water-soluble azo dyes— mono-, bis-, tris-, and tetrakis-azo, selected from the acid, basic, mordant, and direct dyeing classes— have been examined by polarographic methods. The object was to determine how far these methods could be used for the identification and estimation of azo dyes in general. With a few exceptions, the half-wave potential corresponding to the reduction of azo groups does not change very greatly from one dye to another, and this quantity has therefore little value as a general means of identification. When the correct experimental conditions have been established, the polarographic method lends itself to the accurate estimation of all the dyes that have been studied. AUTHORS

Fluorescent Brightening Agents of the Stilbene Series. II— Quantitative Determination of Dinitrostilbenedisulphonic Acid and Dinitrodibenzyldisulphonic Acid
K. Inukai and Y. Maki
Rept. Govt. Ind. Research Inst., Nagoya, **1**, 101–103 (1952):
Chem. Abs., **50**, 3347 (10 March 1956)
Crude dinitrostilbenedisulphonic acid (2 g.) containing the dibenzyl compound is dissolved in water (100 c.c.), acidified with 4% H_2SO_4, neutralised with Na_2CO_3, and brought to 200 c.c. with water. 0·1N-$KMnO_4$ (20 c.c.) is added at 0°c. to 20 c.c. of the above solution, which is

allowed to stand for 2 min., 4% H_2SO_4 (10 c.c.) and KI (2 g.) are added, and the solution is titrated with 0·1N-$Na_2S_2O_3$. 50 c.c. of the original soln. is treated with Fe powder (2 g.) and acetic acid (2 c.c.) on a water-bath for approx. 1 hr., brought to 250 c.c. with water, and titrated with 0·05N-$NaNO_2$ to determine the total amines. The amount of the dibenzyl compound (as Na salt) is given by subtracting the equivalents of iodine from the equivalents of total amines. C.O.C.

Detecting Somalia Red and Mineral Oil Red in Tractor Fuel Oil

M. Palumbo and F. Manfroi

Chimica e Industria (Milan), **37**, 877–879 (1955): *Chem. Abs.*, **50**, 3742 (10 March 1956)

To detect Somalia Red (*o*-tolylazo-*o*-tolylazo-2-naphthol) and similar dyes pass the oil through a column of MgO. Extract the column with glacial acetic acid. A layer of the solution poured into conc. H_2SO_4 indicates presence of the dye by formation of a blue ring at the liquid–liquid interface. Presence of appreciable amounts of unsaturated compounds prevents formation of the blue ring. A more accurate method is to pass 50–100 c.c. of the oil through a column of silica, wash with C_5H_{12}, C_6H_{14}, or methylcyclohexane, with a spatula remove silica above the pink layer, remove this pink layer, extract it with benzene, evaporate to dryness, and dissolve the residue in 50 c.c. of petroleum ether. Alternatively the benzene solution is concentrated, transferred to a Whatman No. 1 filter, dried, and developed with methanol into a yellow–brown area containing impurities from the oil, and a pink or red area containing the dye. This pink area, when treated with alcoholic silico- or phospho-tungstic acid, gives an azure blue ring if Somalia Red or Mineral Oil Red is present. The blue column is stable and obtained with 5–10 μg. of the dye. The test is unaffected by the presence of anti-sludging agents or colour stabilisers, e.g. 2-naphthol, hydroquinone, dialkylphenylenediamine, Chrysoidine Orange Base, Induline, or Nigrosine. Sudan II does not give the blue colour with silicotungstic or phosphotungstic acid. C.O.C.

Petroleum Ether as a Dispersing Agent for Electron Microscopy

E. H. Boult

Nature, **177**, 484 (10 March 1956)

A technique is described in which petroleum ether is used as a dispersing agent for examination of powders by electron microscopy and electron diffraction. The method is suitable for many materials and is rapid, and the material is deposited in a form suitable for shadowing and for electron diffraction. W.R.M.

Instrumental Ranking of Fabric Handle. III—Improvement of Apparatus and Simplification of Test Procedure

H. Uchida, T. Takizawa, and M. Kobayashi

J. Soc. Textile Cellulose Ind. Japan, **12**, 33–40 (Jan. 1956)

Development of an Enzymatic Method for Measuring Changes in Cotton

D. Pesant, R. L. W. Farquhar, and B. A. McLaren

Canadian Textile J., **72**, 33–36 (30 Dec. 1955)

An analytical method is described which uses enzyme hydrolysis as a means of determining changes produced in cotton cellulose or of identifying celluloses of different origins. Using three types— pure cellulose, unpurified cotton fibres, and purified cotton fibres— with Enzyme 19 AP (Rohm & Haas)— a preparation containing cellulase, cellobiase, hemicellulase, gumase, and pentosanase— experimental procedures are defined and tabulated results shown which indicate optimal conditions for each variable in the system. The recommended technique is to incubate a 100-mg. sample of finely ground cellulose in a 15-ml. sintered-glass crucible with 1 ml. of 1% aerosol and 5 ml. of enzyme soln. (6 g./100 ml.) for 5 hr. at 50°c. and pH 4·5. The treated substrate is rinsed, and dried at 105 ± 1°c., cooled, and weighed, and % loss is calculated. A.H.

Determination of Fatty Matter in Scoured Wool and Wool Tops

T. Green, R. P. Harker, and F. O. Howitt

J. Textile Inst., **47**, T 110–T 126 (Feb. 1956)

Methods of determining fatty matter in wool are reviewed, and a systematic series of experiments is made using methylene chloride, ether, ethanol, and benzene–methanol. Soxhlet extraction and the rapid extraction method of Dickinson and Palmer are used, analysis of the extract being confined, by reasons of quantity available, to the former. Differently processed wools— including some almost completely dry— are extracted and results discussed. It appears that conditioned wool gives a higher value than dry. Added (e.g. combing) oil is rapidly removed, and a normal procedure, using ether or methylene chloride, gives an extract containing practically all the added oil. Extraction of soap and grease (wool wax) remaining after scouring depends on the solvent and on the constitution and the distribution of the grease; ether or methylene chloride does not accurately extract the grease, nor does subsequent alcohol extraction accurately evaluate the soap. An initial alcohol extract gives a total extraction value comparable with ether followed by ethanol. An ether-extracted wool will give a further ether extract containing both grease and fatty acid if it is given a thorough acid dip. Solvents of the same general type give similar total extracts, differing mainly in soap and free fatty acid contents. The rapid extraction method with methylene chloride gives a similar total extract to the Soxhlet method using ether. Data found for 64s Australian merino apply generally to other types tested, e.g. crossbreds. Finally, a complete and accurate determination of the fatty matter in wool can be achieved only by a procedure such as exhaustive removal by benzene–methanol followed by analysis of the extract. J.W.B.

Determination of Alkali and Silicate in Peroxide Bleaching Liquors

N. J. Faasen and F. L. J. van Lamoen

Tex, **15**, 188–191 (Feb. 1956)

A rapid volumetric procedure is described for determining the caustic and the silicate contents of peroxide bleaching liquors. The method is based upon the formation of potassium fluosilicate from silica, sodium fluoride, and potassium chloride. Ions such as calcium, magnesium, and iron give no difficulties. If Calgon is present, the caustic content cannot be determined accurately.

AUTHORS

Measuring the Whitening Effects of Fluorescent Brightening Agents and Perborate Bleaches on Cotton

M. S. Furry and P. L. Bensing

Amer. Dyestuff Rep., **45**, P 121 (27 Feb. 1956)

Discussion on paper previously published (*ibid.*, **44**, P 786 (7 Nov. 1955): see J.S.D.C., **72**, 134 (March 1956)).

Effects of Synthetic Detergents on Colour Fastness to Washing

AATCC Washfastness Committee

Amer. Dyestuff Rep., **45**, P 136 (27 Feb. 1956)

When chip soap used in standard test methods for fastness to washing is replaced by an anionic or non-ionic synthetic detergent, with or without the addition of a fluorescent brightening agent, the effect on the colour-fastness rating of the test samples is not significant. Thus, the existing soap formulae are adequate for the purpose of rating the colour fastness of textiles to current laundering practice. J.W.B.

Accelerated Wash-fastness Tests as Suitable End-use Tests for Consumer Goods

AATCC Washfastness Committee

Amer. Dyestuff Rep., **45**, P 134 (27 Feb. 1956)

Classification of Dyes by their Dyeing Characteristics

W. Beal

J.S.D.C., **72**, 146–158 (April 1956)

The methods used for preparing classifications of dyes for application to wool and cotton are reviewed, and particular reference is made to the graphical representation of dyeing characteristics. AUTHOR

Microsublimation of Azoic Bases on a Hot-stage Microscope (IV p. 297)
Coal-tar Colours. XV— FD&C Red No. 1, D&C Red No. 5, and Some Related Colours (IV p. 297)
Application of Conductivity Measurements in Dyeing with Vat Dyes (VIII p. 305)
Application of Weakly Acid-dyeing Metal-complex Dyes (VIII p. 305)
Separation of Formaldehyde during Ironing of Impregnated Fabrics (X p. 308)
Theory of Flame-retardant Finishes (X p. 309)
Cupriethylenediamine as a Solvent for Cellulose (XI p. 310)

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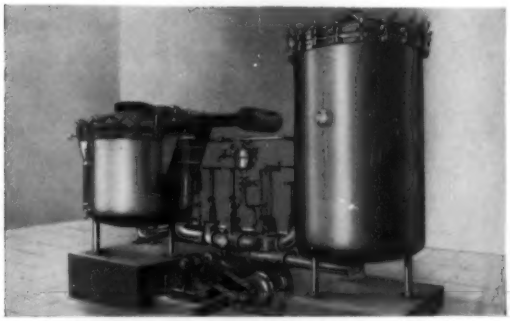

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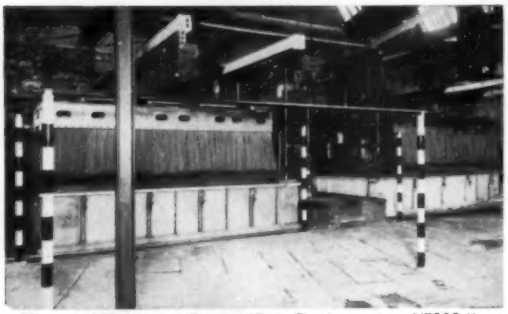

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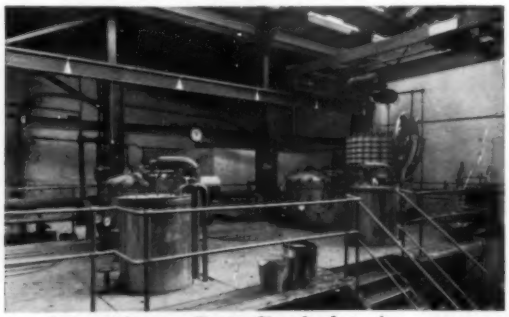

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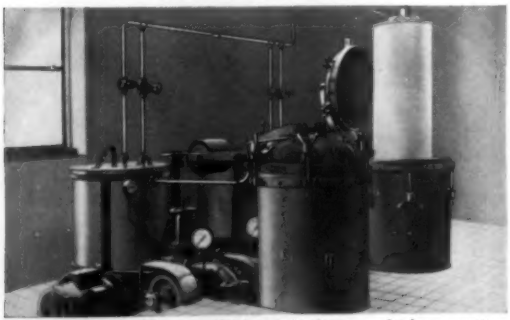

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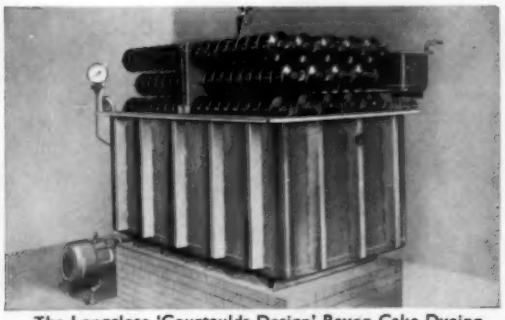

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All inquiries relating to Advertisements in the Journal should be addressed to THE GENERAL SECRETARY, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE.

Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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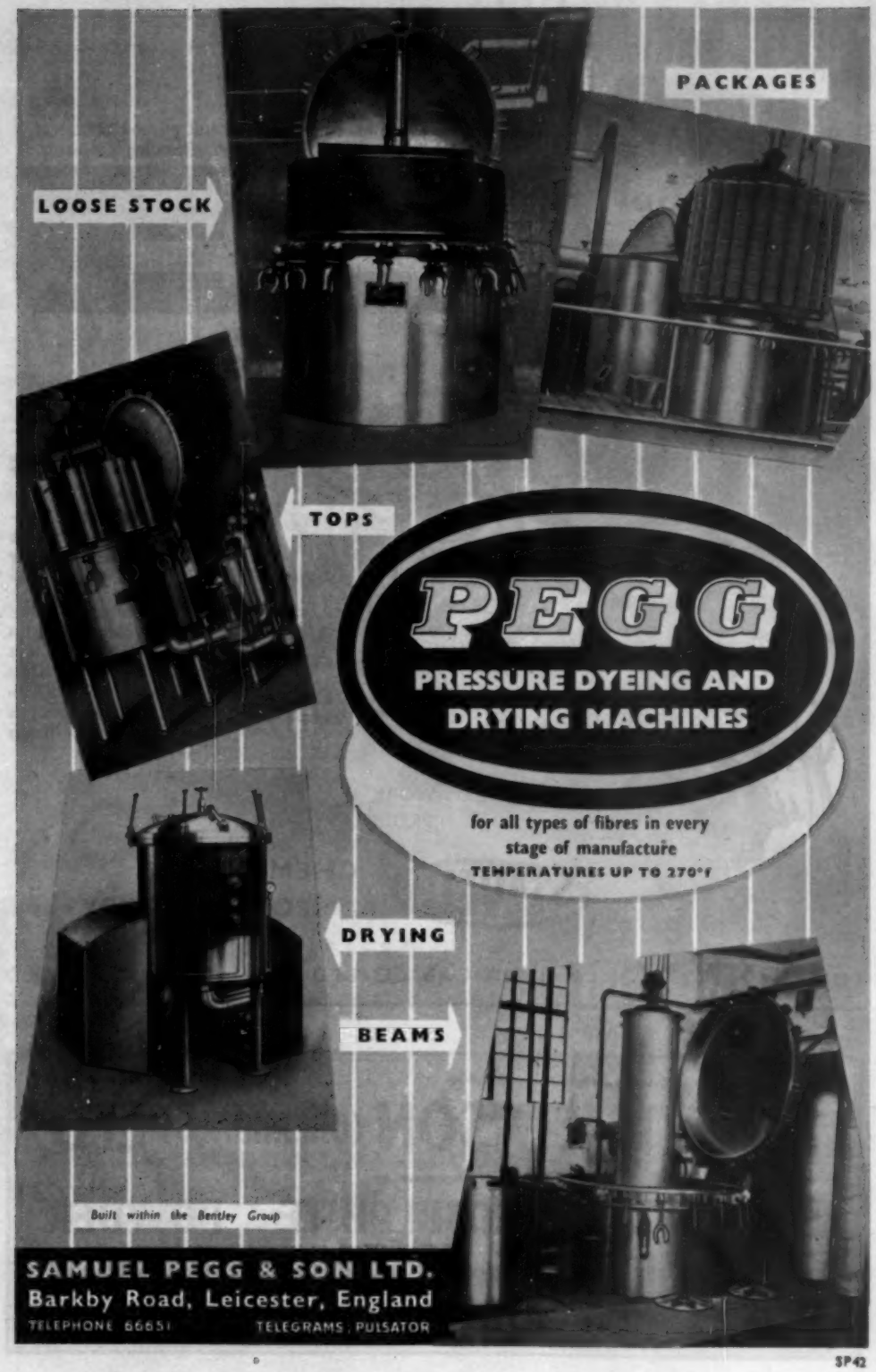
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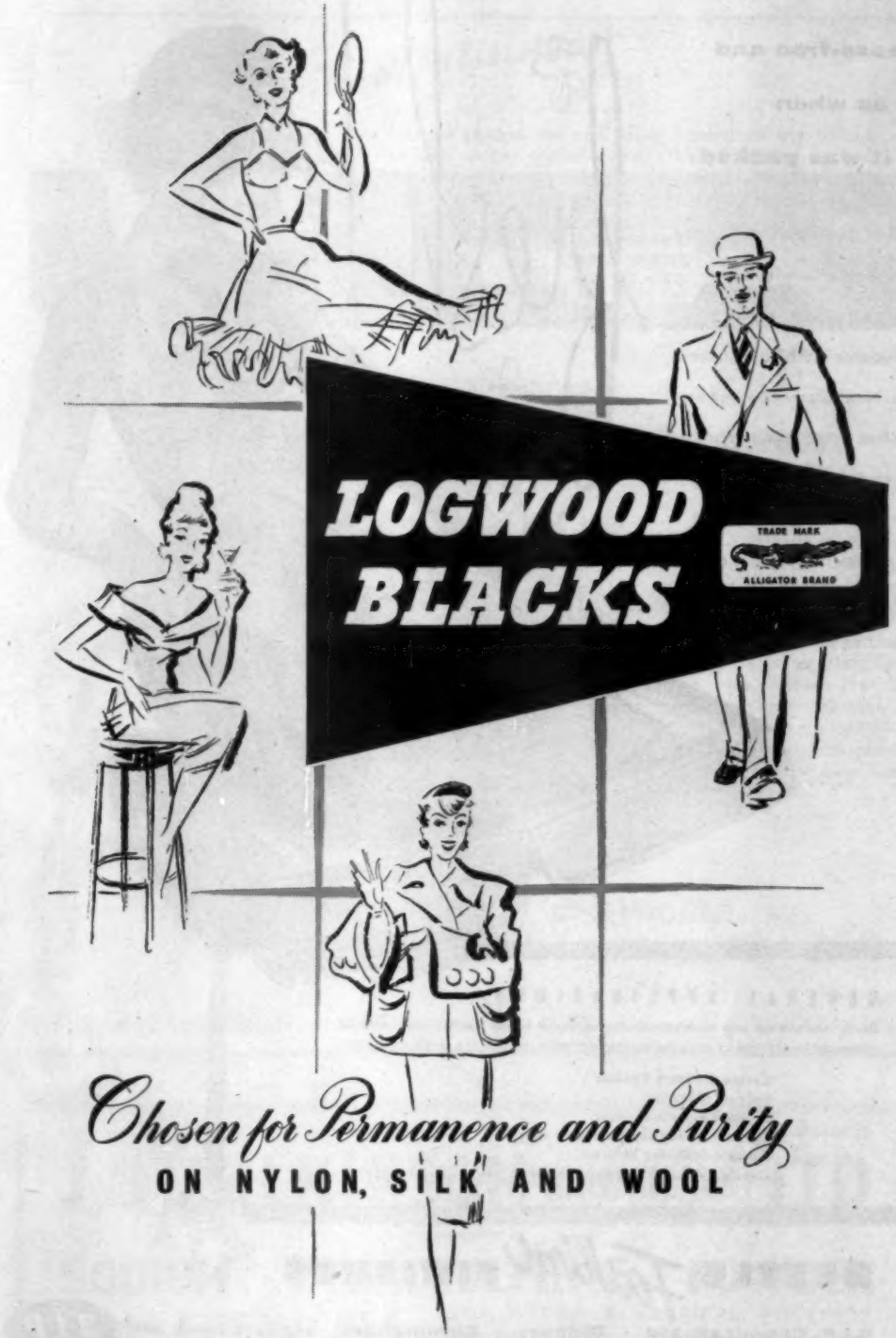

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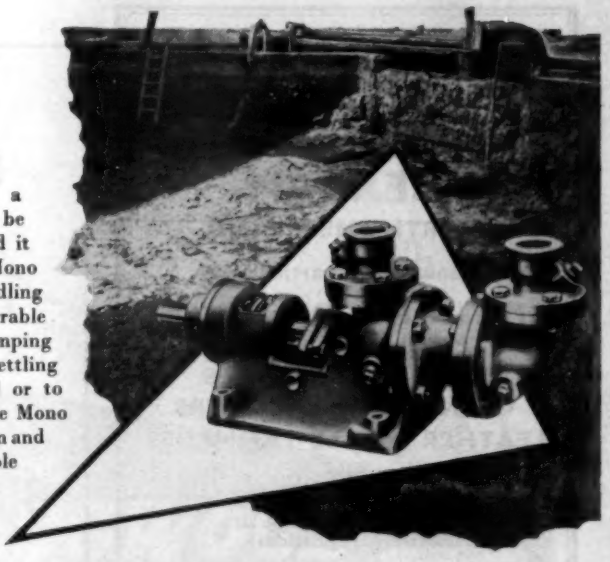

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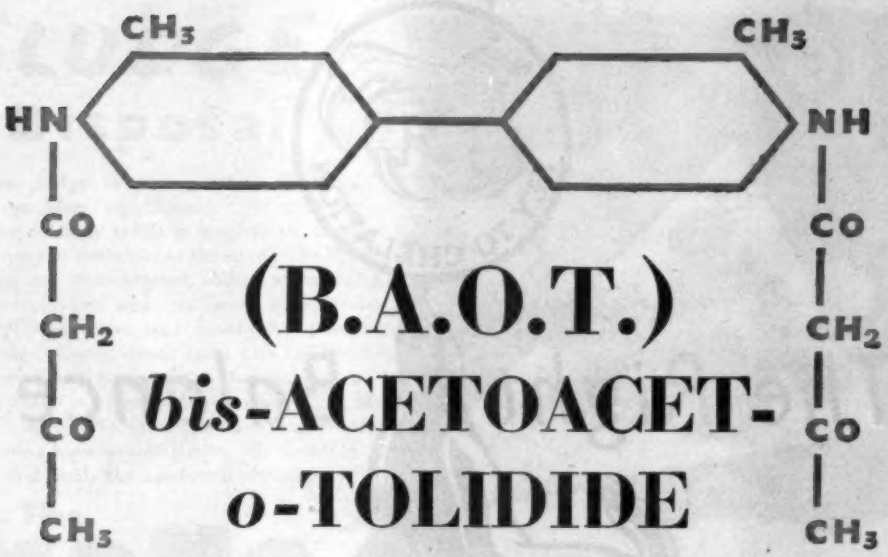

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